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ENVIRONMENTAL CONSULTANTS

FIELD SAMPLING PROCEDURES W. Z. BAUMGARTNER & ASSOC., INC. FRANKLIN, TENNESSEE

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1.0 INTRODUCTION

This document outlines the standard operating procedures (SOPs) and methods — employed by W. Z. Baumgartner & Associates, Inc. to provide quality control of samples collected.

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2.0 GENERAL

2.1 SAMPLE COLLECTION ORDER

Samples shall be collected from the suspected least to the most contaminated sampling locations within a site. Unless field conditions justify other sampling regimens, samples shall be collected in the following order:

- Volatile Organic Contaminants (VOCs)
- Extractable Organics [includes Total Recoverable Petroleum Hydrocarbons (TRPH), Oil & Grease, Pesticides and Herbicides]
- Total Metals
- Dissolved Metals
- Inorganics (Includes Nutrients, Demands and Physical Properties)

2.2 USE OF PROTECTIVE GLOVES

Gloves serve a dual purpose: 1) protects the sample collector from potential exposure to sample constituents; and 2) minimizes accidental contamination of samples by the collector. Protective Gloves will be worn when conducting all sampling protocols, however, their use is not mandatory if:

- The sample source is considered to be non-hazardous; or
- The samples will not be analyzed for trace (i.e. part per billion level) constituents.

If worn, gloves should not come into contact with the sample, the interior of the container or lip of the sample container.

It is recommended that new, disposable, unpowdered latex gloves should be used.

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Gloves should be changed and discarded after every sampling point. Other types of gloves may be used as long as the construction materials do not contaminate the sample or if internal safety protocols require greater protection.

Note that certain materials (as might be potentially present in concentrated effluent) may pass through certain glove types and be absorbed in the skin. There are permeability tables for differing types of gloves that might be advisable in certain situations.

2.3 CONTAINER AND EQUIPMENT RINSING

When collecting aqueous samples the sample collection equipment and non-preserved containers shall be rinsed with sample water before the actual sample is taken.

This protocol shall not be followed for:

- Oil & Grease or TRPH Neither the equipment (if used) nor the container shall be rinsed;
- Microbiological or VOCs Sample containers shall not be rinsed; or
- Sample containers with premeasured preservatives in the container.

2.4 FUEL-POWERED EQUIPMENT

All fuel-powered equipment activities must be placed away from and downwind of any site activities (e.g. purging, sampling, decontamination). If field conditions preclude such placement, (i.e. the wind is from the upstream direction in a boat), the sampling activities shall be conducted as far away as possible from the fuel source(s) and the field notes must describe the conditions.

If possible, fuel handling (i.e. filling vehicles and equipment) should be done prior to the sampling day. If such activities must be performed during sampling, the personnel must

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wear disposable gloves. All fuel dispensing activities and glove disposal shall occur downwind and well away from the sampling activities.

2.5 PRESERVATION

All samples shall be preserved according to the requirements specified in Tables A-2 and A-3 found in appendix A. The holding times listed in the above-referenced tables supersede any that might be discussed in individual analytical methods. The holding times and preservation protocols specified by the tables listed above shall be followed. The preservation protocols in the referenced tables specify immediate preservation. EPA has defined "immediate" as "within 15 minutes of sample collection". This definition shall be followed for all sample preservation. Twenty-four hour composite water samples are the exception to the "15-minute" criteria.

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3.0 DECONTAMINATION

3.1 GENERAL

All equipment shall be cleaned in a controlled environment and transported to the field pre-cleaned and ready to use. All equipment must be immediately rinsed with tap water after use, even if it is to be field cleaned for other sites. Equipment that is only used once (i.e. not cleaned in the field) must be tagged with the sample location, returned to the in-house cleaning facility and cleaned in a controlled environment.

3.2 REAGENTS

Detergents specified in this document refer to Liquinox (or equivalent) or Alconox (or equivalent).

3.2.1 Solvents

The solvent used in routine cleaning procedures shall be pesticide grade or nanograde isopropanol. Pesticide grade and nanograde are synonymous. Other solvents (i.e. acetone or methanol) may be used if they are pesticide grade. However, methanol is more toxic to the environment and acetone may be an analyte of interest for volatile organics. Acetone shall not be used if volatile organics are of interest. Precleaning heavily contaminated equipment may be done with reagent grade acetone and hexane.

3.2.2 Analyte-Free Water

Analyte-free water sources shall be subject to the following criteria:

- Analyte-free water is water in which all analytes of interest and all interferences are below method detection limits.
- This type of water shall be obtained from a reputable source and documentation

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shall be maintained to demonstrate reliability and purity of analyte-free water sources (i.e. results from equipment blanks). As a general rule, the following types of water should be used:

- a. Milli-Q (or equivalent) suitable for all analyses
- b. Organic-free may be suitable for only VOCs and extractable organics
- c. Deionized water suitable for only inorganic analyses (metals, nutrients, etc.)
- d. Distilled water suitable for inorganics or microbiology

Analyte-free water shall always be used for blank preparation and for the final in-house decontamination rinse. Analyte-free water shall be transported to the field in containers of suitable construction.

3.2.3 Protection of Cleaned Equipment

Decontaminated field equipment shall be protected from environmental contamination by securely wrapping and sealing with one of the following:

- Aluminum foil grocery store type is acceptable;
- Untreated butcher paper; or
- Clean, disposable plastic bags may be used if only inorganics are of concern OR the equipment is first wrapped in foil or butcher paper.

3.2.4 Acids

All acids used for cleaning shall be reagent grade or better. Ten percent hydrochloric acid is prepared by mixing one part concentrated hydrochloric acid with 3 parts deionized water. Ten percent nitric acid is prepared by mixing one part

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concentrated nitric acid with 5 parts deionized water.

Prepare acid solutions by slowly adding the concentrated acid to water.

3.3 DECONTAMINATION/CLEANING PROTOCOLS - GENERAL

CONSIDERATIONS

All sampling equipment (bailers, lanyards, split spoons, etc.) that come in contact with the sample must be cleaned/decontaminated before use. The procedures that are applicable to

the majority of sampling equipment are listed in Section 3.4.1 Protocols for other specialized

equipment are outlined in Sections 3.4.2 through 3.11.

If possible, sufficient clean equipment should be transported to the field so that an entire

study can be conducted without the need for field cleaning. Unless otherwise justified, all field

sampling equipment shall be precleaned in-house (office, lab, or base of field operations) prior

to arrival on-site.

All cleaning shall be documented for each piece of field equipment. In-field

decontamination shall be documented in the field records. These records shall specify the type

of equipment that is cleaned and the specific protocols that are used. In-house cleaning records

must identify the type of equipment (i.e. teflon bailers, PVC pump tubing, etc.), the date it

was cleaned, the protocol or SOP that was used and the person who cleaned the equipment.

W. Z. Baumgartner & Associates, Inc. O Environmental Engineers & Consultants O Franklin, Tennessee

3.4 DECONTAMINATION/CLEANING PROTOCOLS - SAMPLING EQUIPMENT

3.4.1 General Cleaning Procedure for Teflon and Stainless Steel Sampling Equipment

This procedure shall be used when sampling for ALL parameter groups: extractable organics, metals, nutrients, etc.) or if a single decontamination protocol is desired to clean all Teflon and stainless steel equipment.

The cleaning procedures described below are for in-field cleaning. Information on in-house cleaning is documented in NOTE 1.

- a. Clean with tap water and lab grade soap (Liquinox or equivalent) using a brush, if necessary, to remove particulate matter or surface film (see NOTES 1, 2 and 3 of this section).
- b. Rinse thoroughly with tap water.
- c. If trace metals are to be sampled rinse with 10-15% reagent grade nitric acid (HNO₃). The acid rinse should not be used on steel sampling equipment (bailers, augers, trowels, etc.). See NOTE 4.
- d. Rinse thoroughly with deionized water (DI). Enough water shall be used to ensure that all equipment surfaces are flushed with water.
- e. Rinse twice with isopropanol. One rinse may be used as long as all equipment surfaces are thoroughly wetted with free-flowing solvent.
- f. Rinse thoroughly with analyte-free water and allow to air dry as long as possible.
- g. Clean sampling equipment shall be wrapped (if appropriate) in aluminum foil, or in untreated butcher paper to prevent contamination

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during storage or transport to the field.

h. If no further sampling is to be performed, equipment must be rinsed with tap water immediately after use.

NOTES:

- 1. In house Protocols require the following:
 - a. Protocols must include the use of HOT tap water and cleaning in a contaminant-free environment.
 - b. Analyte-free water must be used as a final rinse.
- 2. Heavily contaminated equipment should not be cleaned in the field. Such rigorous cleaning procedures should be performed at the base of operations. Cleaning at the base of operations or in the field require the following:
 - a. Pre-rinse equipment using the following solvents in the order described: acetone-hexane-acetone. The solvent rinse(s) must precede the soap and water wash described in the first step (a. above).
 - b In extreme cases, it may be necessary to steam clean the field equipment before proceeding with step a. above.
 - c. If the field equipment cannot be cleaned utilizing these procedures, it should be discarded, unless further cleaning with stronger solvents and/or oxidizing solutions are effective.
- 3. Liquinox (or equivalent) is recommended by EPA, although Alconox (or equivalent) may be substituted if nutrients are not sampled.
- 4. If sampling for nutrients, a 10-15% reagent grade hydrochloric acid (HCl) rinse should be used (except stainless steel equipment). If BOTH metals and nutrients are to be sampled, the HCl rinse must replace the HNO₃ rinse, or the HNO₃ rinse must be followed by the HCl rinse.

- 5. Hot detergent solutions and water rinses are not required for in-field decontamination.
- 3.4.2 Teflon and glass equipment used to collect aqueous and solid samples for trace organics and metals [including oil & grease, TRPH, and total organic halogens (TOX)].

In-house cleaning - follow 3.4.1, see NOTES 1 and 2. In-field cleaning - follow 3.4.1, see NOTES 2 and 5.

3.4.3 Stainless Steel or Other Metallic Equipment used to collect Trace Organics and Metals (includes oil and grease, TRPH, TOX)

In-house cleaning - follow 3.4.1, see NOTES 1 and 2, delete acid rinse. In-field cleaning - follow 3.4.1, see NOTES 2 and 5, delete acid rinse.

3.4.4 All Equipment Used to Collect ONLY Demands and other inorganic nonmetals

In-house cleaning - follow 3.4.1 see NOTE 1, delete solvent and acid rinses.

In-field cleaning - equipment may be rinsed with analyte-free water immediately after use, then rinsed several times with sample water from the next sample.

3.5 FILTRATION EQUIPMENT

- 3.5.1 Dissolved Constituents using In-Line, Molded and Disposable Filter Units
 - a. Peristaltic pump
 - 1. The peristaltic pump is cleaned per section 3.7.2b "Pumps used

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for sampling"

- 2. The silastic pump tubing is cleaned per section 3.6.2
- 3. If Teflon tubing is used, it must be cleaned per 3.6.1
- 4. Other tubing types [high density polyethylene (HDPE), etc.] must be cleaned according to the appropriate protocol listed in 3.6.
- b. Other equipment types (e.g. pressurized teflon bailer)

Other types of equipment that utilize in-line, molded and disposable filters shall follow the appropriate cleaning regimen specified in Sections 3.4.1 through 3.4.6.

3.5.2 Dissolved Constituents using Non-disposable Filtration Units (i.e. syringes, "tripod assembly", etc.)

- a. Proceed with steps a through e of section 3.4.1, assembling and applying pressure to the apparatus after each rinse step (water and acid) to drive rinse material through the porous filter holder in the bottom of the apparatus.
- b. Remove and clean any transfer tubing per appropriate cleaning protocols (see section 3.6).
- c. Assemble the unit and cap both the pressure inlet and sample discharge lines (or whole unit if a syringe) with aluminum foil to prevent contamination during storage.
 - 1. Note: if the unit is to be used to filter only inorganic constituents (i.e. metals, nutrients, etc.), the unit may be sealed in a plastic bag to prevent contamination.

3.6 SAMPLE TUBING DECONTAMINATION

3.6.1 Teflon Tubing

3.6.1.1 New Tubing

If new tubing is used once and discarded, preclean as follows:

- a. rinse outside of tubing with pesticide-grade solvent
- b. flush inside of tubing with pesticide-grade solvent
- c. dry overnight in drying oven or equivalent (zero air, nitrogen, etc.)

3.6.1.2 Reused Tubing

Tubing shall be transported to the field in precut, precleaned sections. The description below was written for in-lab cleaning only, FIELD CLEANING IS NOT RECOMMENDED. In-house cleaning shall follow these steps:

- a. Exterior of the tubing must be cleaned first by soaking the Teflon tubing in hot, soapy water in a stainless steel sink (or equivalent non-contaminating material). Use a brush to remove any particulates, if necessary.
- b. Take a small bottle brush and clean the inside of the tubing ends where the barbs are to be inserted.
- c. Rinse tubing exterior and ends liberally with tap water.
- d. Rinse tubing surfaces and ends with nitric acid, tap water, isopropanol, and finally analyte-free water.
- e. Place tubing on fresh aluminum foil. Connect all of the pre-cut lengths of Teflon hose with Teflon inserts or barbs.
- f. Cleaning configuration:

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- Cleaning reagents (soapy water, acid, isopropanol, etc.) shall be placed in an appropriately cleaned container (2liter glass jar is recommended)
- 2. Place one end of the teflon tubing into the cleaning solution.
- 3. Attach the other end of the teflon tubing set to the INFLUENT end of the peristaltic pump.
- 4. The effluent from the peristaltic pump may be recycled by connecting tubing from the effluent to the glass jar with the cleaning reagents.
- 5. Recycling as described above may be done for all reagents listed in g below **EXCEPT** the final isopropanol rinse and the final analyte-free water rinse. Disconnect the tubing between the effluent end of the pump and the jar of cleaning reagents.
 - a) Isopropanol should be containerized in a waste container for proper disposal;
 - b) Analyte-free water may be discarded down the drain.

g. Using the above configuration:

- 1. Pump copious amounts of hot, soapy water through the connected lengths.
- 2. Follow with tap water, 10% nitric acid, tap water, then isopropanol, and finally analyte-free water.
- During the nitric acid and solvent rinses, turn the pump off and allow the reagents to remain in the tubing for 15 minutes, then continue with the next step. Pumping a

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liter (each) of the nitric acid and solvent should be sufficient, depending on the inside diameter of the hose.

- 4. Leave the Teflon inserts or barbs between the pre-cut lengths and cap or connect the remaining ends.
- h. After the interior has been cleaned as described above, the exterior shall be rinsed with analyte-free water.
- The connected lengths should then be wrapped in aluminum foil or untreated butcher paper and stored in a clean, dry area until use. Documentation for this cleaning shall be noted in the organization cleaning records.

3.6.2 Silastic Rubber Pump Tubing used in Automatic Samplers and other peristaltic pumps

This tubing need not be replaced if the sample does not contact the tubing or if the pump is used for only purging (i.e., not being used to collect samples). Tubing must be changed on a regular basis if used for sampling:

- a. Flush tubing with hot tap water and lab-grade detergent solution
- b. Rinse thoroughly with hot tap water
- c. Rinse thoroughly with DI water
- If used to collected metals samples, the tubing shall be flushed with 1+5 nitric acid, followed by thorough rinsing with DI water
- e. Install tubing in peristaltic pump
- f. Cap both ends with aluminum foil or equivalent

NOTE: Tubing must be changed at specified frequencies as part the preventative maintenance on the equipment.

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3.6.3 Miscellaneous Non-Inert Tubing Types (tygon, rubber, HDPE, PVC, etc.)

3.6.3.1 New Tubing

- a. As a general rule, new tubing may be used without preliminary cleaning.
- b. New tubing shall be protected from potential environmental contamination by wrapping in aluminum foil, sealing in plastic bags or in the original sealed packaging.
- c. If new tubing is exposed to potential contamination, the exterior and interior shall be thoroughly rinsed with hot tap water followed by a thorough rinse with deionized water.
- d. If new tubing is to be used to collect samples, the tubing shall be thoroughly rinsed with sample water (i.e. pump sample water though the tubing) before collecting samples.

3.6.3.2 Reused Tubing

- a. Flush tubing with soapy solution of hot tap water and laboratory detergent.
- b. Rinse exterior and interior thoroughly with hot tap water.
- c. Rinse exterior and interior thoroughly with deionized water.
- d. If used to collected metals samples, the tubing shall be flushed with 10% nitric acid, followed by thorough rinsing with DI water
- e. Wrap tubing and cap ends in aluminum foil and seal in plastic to prevent contamination during storage and transport.

3.7 PUMPS

3.7.1 Submersible pumps

- 3.7.1.1 Pumps used for purging and sampling trace metals and/or organics
 - a. Construction of pump body and internal mechanisms (bladders, impellers, etc.), including seals and connections must follow
 Table A-1 in Appendix A.
 - b. Choice of tubing material must follow Table A-1 in Appendix A.
 - Pump exterior must be cleaned per section 3.4.1.
 Note: the solvent rinse shall be deleted if the pump body is constructed of plastic (i.e. ABS, PVC, etc.)
 - d. Pump internal cavity and mechanism must be cleaned as follows:
 - 1. If for purging only, then the pump must be completely flushed with potable water prior to purging the next well.
 - 2. If for purging and sampling, then it must be completely disassembled (if so designed) and decontaminated between each well.
 - 3. If the pump cannot be (practically) disassembled, then the internal cavity/mechanism must be cleaned by pumping copious amounts of lab-grade soap solution, tap water, and DI water.
 - e. Teflon tubing will be cleaned per section 3.6.1.
 - f. Cleaning of non-inert tubing shall follow the appropriate protocols in section 3.6 above (NOTE: very few options exist for non-inert tubing to be used for purging and/or sampling for trace organics).

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3.7.1.2 Pumps used for all other constituents (nutrient/ inorganic anions)

- a. Pump construction no restrictions
- b. Pump tubing material no restrictions
- c. Scrub the exterior of the pump with appropriate metal-, phosphate- or ammonia-free detergent solution
- d. Rinse the exterior with tap water and deionized water
- e. Rinse interior of pump and tubing by pumping tap or deionized water through the system using clean bucket or drum.

3.7.2 Above Ground Pumps Used for Purging and Sampling

3.7.2.1 Pumps used for purging only

- a. Exterior of the pump must be free of oil and grease
- Tubing choice must follow restrictions as specified in Table A-1 in Appendix A.
- c. Tubing coming in contact with formation water shall be cleaned according to the appropriate protocol for construction materials specified in section 3.6.

3.7.2.2 Pumps used for sampling

- a. Exterior of pump must be cleaned with a detergent wash followed by tap and DI water rinses
- Tubing choice must follow restrictions as specified in Table A-1 in Appendix A.
- c. Tubing coming in contact with formation water shall be cleaned according to the appropriate protocol for construction materials

specified in section 3.6.

3.8 FIELD INSTRUMENTS AND DRILLING EQUIPMENT

3.8.1 Field Instruments (tapes, meters, etc.)

- a. wipe down equipment body, probes, and cables with lab-grade detergent solution,
- b. rinse thoroughly with tap water,
- c. rinse thoroughly with DI water, and
- d. wrap equipment in aluminum foil, untreated butcher paper or plastic bags to eliminate potential environmental contamination.

An optional isopropanol rinse may be performed if equipment comes in contact with contaminated water, etc.

3.8.2 Soil Boring Equipment

This pertains only to equipment that is NOT used to collect samples. Split spoons, bucket augers and other sampling devices must be cleaned per requirements listed in 3.4.1 or 3.4.3 above.

- a. The engine and power head, auger stems, bits and other associated equipment should be cleaned with a power washer, steam jenny or hand washed with a brush using detergent (no degreasers) to remove oil, grease, and hydraulic fluid from the exterior of the unit.
- b. Rinse thoroughly with tap water.

3.8.3 Well Casing Cleaning

These protocols are included and are meant as RECOMMENDATIONS

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for cleaning well casing and riser pipes. Recommendations from other regulatory programs, if different or more stringent shall be followed.

- a. PVC pipe that is designed for well casing shall be transported to the field in original packing boxes.
- b. Other PVC casing (for plumbing, etc. uses) shall be prepared for cleaning by sanding stencils (if present) on those portions of riser pipe that may come in contact with formation water. The ink used for stenciling may contribute to or contaminate the real samples. Casing that has been contaminated with grease, hydraulic fluid, petroleum fuel, etc. may require additional cleaning or deemed unusable.
- c. All casing and riser pipe should be cleaned just prior to installation using the following protocol:
 - 1. Steam clean all casing and riser pipe. Steam cleaning criteria shall meet the following: water pressure 2500 psi, water temperature 200°F.
 - 2. Rinse thoroughly with tap (potable) water. This tap water must be free of the analytes of interest, in effect analyte-free. Some potable water sources may have very low levels of contamination (e.g. benzene, trihalomethane, trichloroethane). A potable water source should only be used if it is known to be free of those contaminants that are being investigated.

3.9 ANALYTE-FREE WATER CONTAINERS

Analyte-free water containers made be constructed of glass, Teflon, polypropylene, or high density polyethylene (HDPE). It is strongly recommended that inert glass or Teflon be used for containerizing organic-free sources of water.

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Polypropylene is a good second choice. HDPE, though not recommended, is acceptable. Analyte-free water should not be left in these containers for extended periods, especially HDPE. These containers should be filled up for a single sampling — event and then emptied at the end of the sampling day. EPA's cleaning procedure for glass (Teflon and polypropylene) is as follows:

3.9.1 New Containers

- a. Wash per instructions in 3.4.1 (delete solvent rinse if plastic (HDPE or polypropylene) containers are being cleaned).
- b. Cap with Teflon film, aluminum foil or the bottle cap. Note: the bottle cap shall be equipped with a teflon liner. Aluminum foil or teflon film may be used as liner material.

3.9.2 Reused Containers

- a. Immediately after being emptied, cap with aluminum foil, teflon film or the container cap.
- b. Wash container exterior with lab-grade detergent solution and rinsed with DI water
- c. Rinse interior twice with isopropanol (delete if containers are plastic, see 3.9.1a above)
- d. Rinse interior thoroughly with analyte-free water,
- e. Invert and allow to drain and dry
- f. Fill container with analyte-free water and cap tightly with aluminum foil, Teflon film or the container cap. Note: the bottle cap shall be equipped with a teflon liner. Aluminum foil or teflon film may be used as liner material.
- g. Water shall not be stored for more than 3 days prior to a sampling trip.

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3.10 ICE CHESTS AND SHIPPING CONTAINERS

- a. Wash exterior and interior of all ice chests with laboratory detergent (see 3.4.1 NOTE 3).
- b. Rinse with tap water and air dry before storage.
- c. If the ice chest becomes severely contaminated with concentrated waste or other toxic or hazardous materials, it should be cleaned as thoroughly as possible, rendered unusable, and properly disposed.

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4.0 AQUEOUS SAMPLING PROCEDURES

4.1 General

There are several requirements that are common to all types of surface water sampling events and are independent of technique. Several of these requirements are concerned with sample parameters that are inherently difficult to sample. In addition to the below procedures, overall care must be taken in regards to equipment handling, container handling/storage, decontamination, and record keeping.

- a. Sample collection equipment and non-preserved sample containers must be rinsed with sample water before the actual sample is taken. Exceptions to this are: oil & grease, TRPH, microbiological, VOCs, or any pre-preserved container.
- b. If protective gloves are used, they shall be clean, new and disposable. These should be changed prior to moving to the next sampling point.
- c. Sample containers for source (i.e. concentrated wastes) samples or samples suspected of containing high concentrations of contaminants shall be placed in separate plastic bags immediately after collecting, preserving, tagging, etc.
- d. If possible, ambient, or background samples should be collected by different field teams. If separate collection is not possible, the ambient or background samples shall be collected first and placed in separate ice chests or shipping containers. Highly contaminated samples shall never be placed in the same ice chest as environmental samples. It is a good practice to enclose highly contaminated samples in a plastic bag before placing them in ice chests. Ice chests or shipping containers with samples suspected of being highly contaminated shall be lined with new, clean, plastic bags.
- e. If possible, one member of the field team should take all the notes, fill out tags,

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- etc., while the other member does all of the sampling.
- f. Teflon or glass is preferred for collecting samples where trace contaminants are of concern. Equipment constructed of rubber or plastic (e.g., PVC, Tygon, most Van Dorn Samplers) shall not be used to collect samples for trace organic compound analyses.

4.2 Special Parameter - Specific Handling Procedures

- a. Since the concentration standards and/or guidance criteria for many analytes are in the (sub)parts per billion range, extreme care must be taken to prevent cross-contamination.
- b. Most of the parameter groups listed in sections 4.2.1 through 4.2.5 below, shall be taken as grab samples unless regulatory requirements dictate otherwise. The exceptions are extractable organics and total metals which may be taken as composites, if required.
- c. There is a greater chance of cross contamination when collecting composites because of increased sample handling and more equipment.
- d. The following eight categories of parameters have specific sampling techniques and considerations which must be followed to collect unbiased, uncontaminated samples.

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THE PROCEDURES OUTLINED BELOW SHALL BE USED FOR ALL AQUEOUS SAMPLING (I.E. SURFACE WATER, GROUNDWATER, ETC.).

4.2.1 Metals Sampling

4.2.1.1 Sample containers

- a. New or properly cleaned plastic containers may be used for metals sampling. Glass bottles may also be used, but they are prone to breakage and occasionally react with the sample to either leach or adsorb metals from the glass itself.
- b. All containers for metals sampling, new or previously used, shall be cleaned by following protocols outlined in Section 6.1.
- c. Visually inspect polyethylene or glass containers for defects or contamination. Discard if defects are present or containers do not appear clean.

4.2.1.2 Preservation

- a. Samples shall be preserved with nitric acid (HNO₃) of a grade that is suitable for use in trace metals analysis.
- b. Preservation shall occur within 15 minutes of sample collection or filtration (if applicable) unless collected as a 24-hour composite.
- c. Adequate HNO₃ shall be added per liter of sample to reduce the pH to below 2.0 to keep metals in solution and prevent them from adsorbing or absorbing to the container wall.
- d. If only dissolved metals are to be measured, the sample shall be filtered immediately after sample collection through a 1.0 μm membrane filter for groundwater and a 0.45 μm membrane filter

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for surface water. The sample shall not be preserved before filtration. See Table A-1 for approved filtration equipment

4.2.1.3 Sample collection protocol:

- a. Remove the cap from the sample container and rinse container with sample water (IF NOT PRE-PRESERVED). Carefully pour sample into the container without allowing the sampling device to touch the rim of the sample container.
- b. If adding preservatives in the field, the sample container should not be filled to capacity.
- c. Acidify the sample to pH of 2 or less by adding a measured quantity of concentrated HNO₃ or 1+1 HNO₃ into the container.
- d. NOTE: If containers are pre-preserved by a subcontract laboratory, the sample must be poured into the container slowly to prevent the acid from splattering. As a precautionary note, the addition of water to acid can generate enough heat to burn unprotected hands.
- e. Tightly cap the sample container and shake to distribute the acid. Pour an aliquot of the acidified sample into a disposable container (e.g. sampling cup) or onto a piece of NARROW range pH paper to determine if the pH is less than 2.0. DO NOT PUT THE pH PAPER DIRECTLY INTO THE SAMPLE CONTAINER!
 - Field experience has shown that UNDER NORMAL CIRCUMSTANCES, 2 ml of concentrated HNO₃ added to 250 ml of sample water will reduce the pH to less than 2.

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- 2. If the pH is greater than 2, add additional MEASURED amounts of acid until the pH has been reduced.
- 3. Record the total amount of acid that was added to the sample. This documentation is necessary for the next site visit, since additional acid may need to be added to the sample on subsequent visits.
- 4. Acidify at least one of the equipment blank(s) with the GREATEST amount of acid that was required in the sample set and note the amount in field documentation.
- f. Following proper sample preservation, tightly cap, affix a sample label, apply a seal (if required), and complete the chain of custody.
- g. Aqueous samples for metals need not be cooled to 4° C.
- h. Make a note on the transmittal form identifying samples that have entrained sediment.

4.2.1.4 Filtration

- a. All samples that are filtered shall be identified in field notes and on final reports as "dissolved" or "filtered" metals.
- b. Specific protocols for collecting dissolved metals from groundwater samples are discussed in Section 4.4.6.4.
- specified for groundwater (Section 4.4.6.4) These protocols are recommended when sampling static surface water sources (i.e. subsurface samples from lakes, ponds, or lagoons) since exposure to air can change the concentration of metals in solution. When sampling from moving sources (i.e. rivers or

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streams) or just below the surface, filtered samples may be collected into an intermediate container and filtered with syringe-type or tripod type filtration units.

- d. Allowing a sample to settle and decanting the supernate (upper water layer) has been proposed as a means of removing suspended material. This technique MAY NOT be used for groundwater samples, and is not recommended for other sources because:
 - 1. Settling times techniques are highly dependent on particle size and concentration and may not be reproducible;
 - Preservation for metals must occur within 15 minutes of sample collection which may not be sufficiently long for highly turbid samples to settle; and
 - 3. The analytical results cannot be reported as "total" or "dissolved".

If this technique is used, the following protocols must be followed:

- 1. Samples shall not be acidified before settling occurs;
- Total time for settling shall not exceed 15 minutes;
- The resultant supernate shall be carefully decanted into an appropriate container and preserved using protocols described above;
- 4. Field notes shall specify the length of time the sample was allowed to settle, as well as observations on the initial and final (supernate); and
- 5. The final report shall identify the technique that was used to collect the sample (i.e. decanted).
- 6. NOTE: samples SHALL NOT be transported back to the

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laboratory for settling, UNLESS entire procedure (transport, settling, decanting and preservation) can occur within 15 minutes of sample collection.

4.2.2 Extractable Organics and Pesticides

Conventional sampling practices shall incorporate the following special considerations. Oil & Grease (O&G) and Total Recoverable Petroleum Hydrocarbons (TRPH) shall follow protocols outlined in Section 4.2.4 below.

4.2.2.1 Sample containers

- Collect all samples in glass containers (1 liter to 1 gal.) with
 Teflon-lined caps. Note: Teflon containers are also acceptable.
- b. Amber glass should be used for PAHs.
- c. Visually inspect glass bottles to assure that there are no glass or liner defects. If defects are present and/or the sample containers do not appear clean, the bottles must be discarded.
- d. Sample containers must be cleaned according to the protocols specified in Section 6.1.

4.2.2.2 Preservation

- a. Table A-2 must be followed to determine the specific preservation method for each group of organic compounds and pesticides.
- All samples must be placed on wet ice immediately after collection. Samples must be maintained at a temperature of 4°
 C.
- c. If the samples for pesticides cannot be extracted within 72 hours

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of collection, the sample pH must be in the range of pH 5 to 9. If needed, sample must be adjusted to the specified pH range with sodium hydroxide or sulfuric acid.

- d. Other extractable samples need not be pH-adjusted with acid or base.
- e. Samples must be extracted within 7 days of sample collection and the extracts analyzed within 40 days of extraction.

4.2.2.3 Sample collection protocol:

- a. Sample bottles should be prerinsed with sample before collection, except Total Recoverable Petroleum Hydrocarbons (TRPH), Oil & Grease, etc. or any prepreserved sample container.
- b. Remove the cap from the bottle without touching the Teflon liner.
- c. Do not allow the sampling equipment or hands to touch the rim of the sample container.
 - 1. For bailer sampling, it may be necessary to utilize a stainless steel or Teflon delivery tube (fits into the bottom of the bailer).
- d. Fill bottle with sample to almost full capacity.
- e. Quickly place the Teflon lined cap over the bottle and tighten securely.
- f. Affix a sample label, seal (if required), and complete the chain-of-custody form.
- g. Put the sample bottle in a plastic sample bag and place on wet ice immediately.

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h. Make a note on the lab transmittal form identifying samples that appear highly contaminated or exhibit other abnormal characteristics (i.e. foaming, odor, etc.).

4.2.3 Volatiles Sampling

4.2.3.1 Sample containers

- a. Analysis of volatile organic substances requires a glass sample vial, sealed with a teflon-coated septum.
- b. AT A MINIMUM, duplicate samples must be collected, although some laboratories require three or more vials. If the containers are not supplied by the laboratory, verify the laboratory's policy on how many vials are necessary and collect the specified number of vials.
- c. Visually inspect the glass vials to assure that there are no glass or septum defects (e.g. rim must have not nicks or visible depressions); septum must not be deformed, etc.). If defects are present and/or sample containers or septums do not appear to be clean, the vials must be discarded.
- d. Sample vials may be purchased precleaned from commercial vendors, or must be cleaned according to protocols outlined in Section 6.1.
- e. NOTE: VIALS FOR VOCS ARE NOT RINSED WITH SAMPLE.

4.2.3.2 Preservation

a. Table A-2 must be followed to determine the specific preservation method for each group of volatile organic

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compounds.

- b. The vials shall be filled with the sample, acidified (prepreserved containers are acceptable) with HCl and labeled "preserved".
- c. If the volatile aromatics are to be analyzed within 7 days, HCl is not necessary.
- d. Samples must be placed on wet ice immediately after sample collection. A temperature of 4°C must be maintained until the sample has arrived at the laboratory.

4.2.3.3 Sample collection protocols:

- a. All fuel or exhaust sources which could cause VOC contamination must be situated well away and downwind of the sampling site.
 - 1. If possible, fuels should be transported and stored in a separate vehicle from empty vials and collected samples.
 - 2. All petroleum fueled engines (including the vehicle) must be situated downwind of the sampling operations.
- b. Samples shall not be aerated during sample collection.
 - 1. Extreme caution must be exercised when filling a vial to avoid any turbulence which could promote volatilization.
 - 2. Carefully pour the sample down the SIDE of the vial to minimize turbulence. As a rule, it is best to gently pour the last few drops into the vial so that surface tension holds the water in a "convex meniscus."
- c. Do not allow the sampling equipment to touch the rim of the sample container.
 - 1. For bailer sampling, it may be necessary utilize a

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- stainless steel or Teflon delivery tube or "pigtail" to obtain a gentle trickle of sample into the vial.
- 2. It is sometimes difficult to completely fill the vial directly from some waste streams. The sample may be collected in a precleaned intermediate sample collection device made of the appropriate materials (see Table A-1) and carefully poured into the VOC vials.
- d. The sample must be collected so that there are no air bubbles in the container after the screw cap and septum seal are applied.
 - 1. Vial must be filled so that the sample surface is above the container rim (convex meniscus).
 - 2. The cap with the septum is then quickly applied (make sure teflon side of septum is down). Some sample may overflow, but air space in the bottle must be eliminated.
 - 3. If acid has been added to the sample, tip the vial gently two or three times to distribute the preservative.
 - 4. Turn the bottle over and tap it to check for bubbles. If any are present, remove the cap, add a few more drops of sample, recap and test for bubbles. REPEAT NO MORE THAN 3 TIMES.
- e. Sampling and preservation containers may be prelabeled prior to any field activities. This may reduce confusion during a sampling event.
- f. All the vials must be labeled. Make note in the field records of any samples that appear highly contaminated or appear to effervesce when acid is added.
- g. Wrap each vial in bubble-wrap, or equivalent, and place each

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vial in a small ziplock-type bag and immediately place on wet ice.

- h. Complete field records.
- i. Protect samples from environmental contamination during storage and transport to the laboratory.
 - 1. As an added measure, replicate samples may be sealed in a container with vermiculite. This will add further protection from potential contamination.

4.2.4 Oil and Grease (O&G) and Total Recoverable Petroleum Hydrocarbon (TRPH) Sampling

4.2.4.1 Sample Containers

- Samples for O&G and TRPH shall be collected in 1 liter widemouth glass bottles.
 - 1. The lid shall be teflon-lined.
 - 2. If the cap is not teflon-lined, a sheet of teflon extending out from the lid may be used.
- b. Visually inspect glass bottles to assure there are no glass or cap defects. If defects are present and/or sample containers do not appear to be clean, the bottles should be discarded.

4.2.4.2 Sample Preservation

- a. Since losses of the product will occur on sampling equipment, composite samples shall not be collected.
- b. The sample must be immediately preserved by adding H_2SO_4 or HCl to reduce the pH to 2.0 or less.
- c. Samples must be placed on wet ice immediately after

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preservation. The temperature of the sample must be maintained at 4°C until received and processed by the laboratory.

4.2.4.3 Selection of Sampling points

- a. Oil and grease may be present in wastewater as a surface film, an emulsion, a solution, or as a combination of these forms. Since it is very difficult to collect a representative ambient sample for oil and grease analysis, the sampler must carefully evaluate the location of the sampling point.
- b. The most desirable sampling location for both O&G and TRPH is the point where greatest mixing is occurring. Quiescent areas should be avoided, if possible.
- c. Skimming the surface for the sample is unacceptable.
- d. For compliance samples at a facility you may want to take samples at the worst place.
- e. Neither the container, nor the sampling device, shall be rinsed before the actual sample is taken.
- f. COMPOSITE SAMPLES SHALL NOT BE COLLECTED. If composite data is required, individual grab samples that are collected at prescribed time intervals must be analyzed separately to obtain the average concentrations over an extended period.

4.2.4.4 Sampling Protocols

- a. Sampling for these products is unique because they are immiscible and tend to adhere to the sampling device; therefore, these sample shall always be a grab sample.
- b. The sample, when collected, should not be transferred to another

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container. The analytical methods require the use of the entire sample. In addition, the sample container must be rinsed with solvent as a part of the laboratory analytical process. Therefore these samples must be separate and discrete samples that will be used only for the O&G or TRPH analysis.

- c. Remove the cap from the glass bottle without contacting the interior of the container or lid.
- d. DO NOT RINSE THE BOTTLE WITH SAMPLE WATER.
- e. Whenever possible samples should be collected directly into an unpreserved sample container. If intermediate sampling equipment is used, do not allow the sampling equipment to touch the rim of the sample container. AUTOMATIC SAMPLERS SHALL NOT BE USED TO COLLECT THESE TYPES OF SAMPLES.
- f. Fill the bottle with the sample water to almost full capacity.
- g. Add preservatives and check the pH using the protocols outlined in 6.2.2.b.
- h. Quickly cap the container and tighten securely.
- i. Affix a sample label, seal (if required), and complete the chain-of-custody form.
- j. Protect glass container from breakage ("bubble wrap" is recommended), place the sample bottle in a plastic sample bag and keep it cool to 4°C on wet ice.
- k. Make a note on the lab transmittal form identifying samples that may be highly contaminated or any other unusual observations.

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4.2.5 Cyanide Sampling

Cyanide is a very reactive and unstable compound. Cyanide should be analyzed as soon as possible after collection.

4.2.5.1 Sample Containers

- a. The sample container shall be polyethylene or glass.
- b. Containers shall be cleaned in accordance with protocols outlined in Section 6.1 of this manual.

4.2.5.2 Preservation

- a. All samples shall be preserved to a pH of greater than 12 with sodium hydroxide and placed on wet ice immediately after preservation. A temperature of 4°C shall be maintained until analysis begins at the laboratory. The pH of the samples shall be checked to assure proper pH (see 6.2.2.b).
- b. All samples known to contain oxidizing agents (chlorine) must first be tested as follows:
 - 1. Test sample with KI-starch paper;
 - 2. Add a few crystals of ascorbic acid, mix sample and retest.
 - 3. Continue to add ascorbic acid until the test is negative;
 - 4. Add an additional 0.6 grams of ascorbic acid per liter of sample to remove chlorine.

4.3 Surface Water Sampling

4.3.1 Introduction and Scope

This section presents the standard operating procedures that shall be

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employed during field investigations to ensure that representative surface water samples are collected. The particular surface water types that will be addressed include; static lakes, ponds, and impoundments; tidally-influenced estuarine areas; as well as streams and rivers. Care should be taken so that samples are neither altered nor contaminated by sample handling procedures.

This section discusses grab, depth-specific, and depth composited surface water samples. Information regarding flow- or time-weighted aqueous sampling is found in the Wastewater Sampling section.

4.3.2 General

Access will be left up to the sampling group. Ease of access should not be the main criteria for sampling site choice. If sampling from a bridge, by boat, or by wading, there are certain precautions that must be considered:

- a. If sampling with a boat, samples should be taken from the bow, away and upwind from any gasoline outboard engine.
- b. Collect samples upstream from the body when wading in to collect water samples.
- c. Care should be taken not to disturb sediments when taking samples in lakes, ponds, impoundments.
- d. If water samples and sediment samples are to be taken from the same area, the water samples must be taken first.
- e. Sampling at or near structures (dams, weirs, bridges) may not provide representative data because of unnatural flow patterns.
- f. Surface water and/or sediments should always be collected from downstream to upstream.

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4.3.3 Sample Acquisition

Three (3) types of general sample acquisition methods will be discussed: grab samples; mid-depth samples; and composite samples.

4.3.3.1 Grab Sampling

- a. If the sample media is homogenous, grab samples are an effective and simple technique. If homogeneity is not known (and should never be assumed) then other techniques must be used.
- b. Surface grabs using unpreserved sample containers are encouraged since the sample container is used for collecting the sample and, after appropriate preservation, the same container can be submitted for laboratory analysis. This reduces sample handling and eliminates potential contamination from other sources (i.e. additional sampling equipment, environment, etc.). If the laboratory provides prepreserved sample containers, the sample shall be collected in an UNPRESERVED sample container or with sampling equipment. The container or equipment shall be of appropriate construction (see Table A-1) and the sample shall be transferred immediately into the prepreserved sample container.
- c. Simple Grab Samples Typical sample collection equipment includes not only sample containers, but also precleaned beakers, buckets, and dippers. These samplers must be constructed appropriately (including handles):
 - 1. Sample Container (unpreserved)
 - a) submerge the container, neck first into the water,

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- b) invert the bottle so the neck is upright and pointing into the water flow (if applicable),
- c) return the filled container quickly to the surface,
- d) shake to rinse the interior surface of the container and pour contents out downstream of sample location (see restrictions outlined in 2.3)
- e) Collect sample as described in steps a, b and c above.
- f) pour out a few mls of sample downstream of sample collection. This allows for addition of preservatives and sample expansion
- g) Securely cap container, and label.

2. Intermediate vessel

- a) Collect sample as outlined in C.1. above.
- b) Pour into prepreserved sample container (or field preserve per Section 6.2.2.a), check pH per Section 6.2.2.b (if applicable), cap, and label.
- d. Pond Sampler Another effective technique is using a polemounted flask, beaker, or container. A long, telescoping pole (swimming pool supply) is outfitted with a (non-contaminating) clamp. An appropriately constructed and shaped container is fitted into the clamp. In this way the sample can be taken away from the shore, boat, bridge, etc. and at a specific spot. The sampling vessel can be constructed of all-inert material so that all parameters can be sampled.
 - 1. Submerge the clamped container neck first, invert and withdraw from water.

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- 2. Be careful not to entrain sediments or skim the water surface.
- 3. Rinse container (restrictions specified in Section 2.3 must be observed), resubmerge and collect sample. Retrieve the pole, clamp, and container and fill the sample containers.
- e. Pump and Tubing Although the use of a peristaltic pump and tubing can provide an adequate mid-depth or depth composite, it can also be used for taking a grab sample. This would be especially helpful if a large amount of sample is needed.
 - 1. Lower appropriately precleaned tubing to a depth just below the water surface (6 12 inches).
 - 2. Turn the pump on.
 - 3. Allow several tube volumes to pump through the system to acclimate the tubing.
 - 4. Make sure the tubing does not come out of the water and inadvertently pull some surface skim water through the tubing (this may bias sample results).
 - 5. Fill the individual sample bottles via the discharge tubing.

NOTE: THIS TECHNIQUE IS NOT ACCEPTABLE FOR OIL & GREASE, TRPH OR VOCs. It is not recommended for extractable organics or microbiologicals (new, unused tubing, including tubing in the sampling head are required at each sampling location).

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4.3.3.2 Mid-Depth Sampling

- a. Mid-depth samples or samples taken at a specific depth can approximate the conditions throughout the entire water column.
 - 1. One sample may be taken when the water body is assumed to be homogenous.
 - Additional samples can be taken from different depths at one spot to get a much more exact approximation of the conditions.
 - 3. Many times a single site will be sampled from: just below the surface; mid-depth; and just above the bottom (sediment).
 - 4. Accurate sampler location is imperative for this sampling technique.
- b. The equipment that may be used for this type of sampling are:
 a device designed specifically for depth-specific sampling
 (kemmerer, niskin, beta, etc.); pumps with tubing; or double
 check valve bailers.
 - 1. Samplers are available from many manufacturers and in a variety of configurations and construction materials.
 - 2. Certain construction material details may preclude a device's use for certain parameters (see Table A-1):
 - a) Many kemmerer samplers are constructed of plastic and rubber which precludes their use for all organic sampling parameters (volatile and semivolatile).
 - b) Some of the newer devices are constructed of stainless steel or are all-Teflon or Teflon coated.

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These would be acceptable for all parameter groups without restriction.

- c) NOTE THAT ALL RELATED COMPONENTS
 (STOPPERS, ETC.) MUST BE
 CONSTRUCTED OF INERT MATERIAL AS
 WELL IF ORGANICS ARE TO BE SAMPLED.
- c. Kemmerer, niskin, and beta type devices
 - Separate and specific deployment discussions are not provided in this document. Manufacturers suggestions shall be followed for specific procedures.
 - a) Before lowering the sampler, measure the water column to determine maximum depth and sampling depth.
 - b) The line attached to the sampler should be marked with depth increments so that the sampling depth can be accurately recorded.
 - c) When dropping the sampler to the appropriate depth, it should be done slowly so that sediments are not stirred up.
 - d) Once the desired depth is reached, send the messenger weight down to trip the mechanism.
 - e) The sampler should be lowered and retrieved slowly.
 - f) The first sample shall be discarded into a bucket (to be dumped at conclusion of sampling).
- d. Double check-valve bailers
 - 1. Sampling with these type of bailers shall follow the same

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protocols outlined in "c" above.

- Although not designed specifically for this kind of sampling, it will be acceptable when a mid-depth sample ____ is required.
- 3. Note: this sampler does not perform as well as the devices described above or the pump and tubing described in the next section.
- 4. As the bailer is dropped through the water column, water will be displaced through the body of the bailer. The degree of displacement is dependent upon the check valve ball getting out of the way and allowing water to flow freely through the bailer body.
- 5. An open-top bailer may also be used, but is not recommended. The open-top arrangement will not prevent water from being exchanged in the top portion of the bailer.
- 6. A closed-top bailer does not allow free water displacement on descent at all and is not acceptable.
- 7. The bailer should be dropped slowly to the appropriate depth. Upon retrieval, the (two) check valves seat, preventing water from escaping out of or entering the bailer.

e. Pump and Tubing

- 1. The most portable pump for this technique is a (12 volt) peristaltic pump.
- 2. Appropriately precleaned silastic is required in the pump head and HDPE, Tygon, etc. tubing is attached to the

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pump.

- 3. Measure the water column to determine the maximum depth and the sampling depth.
- 4. Tubing will need to be tied to a stiff pole or be weighted down so the tubing placement will be secure.
 - a) A lead weight should not be used.
 - b) Any dense, non-contaminating, non-interfering material will work (brick, SS weight, etc.).
 - c) Tie the weight with a lanyard (braided or monofilament nylon, etc.) so that it is located below the inlet of the tubing.
- 5. Turn the pump on and allow several tubing volumes of water to be discharged before taking the first sample.
- 6. Sample containers are then filled in the proper order, preserved, labeled, and placed on ice (if required).

4.3.3.3 Composite Sampling

Composite sampling will be used when a single sample that approximates a given depth interval is desired. Any of the devices described in mid-depth sampling can be used for composite sampling. The devices must be activated or manipulated in a way that the actual volumes sampled within the interval are ALL EQUAL PROPORTIONS. For instance, because of head pressure, the pump and tubing will pull a greater volume of sample at 5 feet in comparison to 20 feet. For this reason, great care must be used so that sample results are not biased. The use of the niskin, kemmerer, beta, bailers, etc. containers may take more time, but sample control will be greater.

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4.4 Groundwater Sampling

4.4.1 Introduction and Scope

This section presents the standard operating procedures that should be employed during field investigations to ensure that representative groundwater samples are collected. Care should be taken so that the sample collected is neither altered nor contaminated by sampling and handling procedures.

The following discussions cover acceptable: equipment choice, equipment construction materials, pre-sampling and in-field decontamination, purging and sampling technique, and proper field Quality Control procedures. Although not a complete discussion of all groundwater sampling procedures, this information has been compiled with the intent of providing the equipment and techniques for situations that are most likely to be encountered.

4.4.2 Purging and Sampling Equipment

4.4.2.1 General Considerations

- a. Purging the monitor well of stagnant water can be performed with various equipment. The choice of equipment will depend on the parameters of interest, the well diameter, the well specific capacity, transmissivity, the water level elevation and other site conditions. As stated earlier, the choice of equipment used for purging must not bias the "representativeness" of the sample collected.
- b. It is recommended that field personnel use pumps to purge monitor wells if at all possible.
- c. Bailers are not recommended for purging monitor wells because frequent lowering and retrieving of the bailer:
 - 1. will introduce atmospheric oxygen which may precipitate metals

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- (e.g. iron) or cause other changes in the chemistry of the formation water (i.e. pH),
- 2. will result in agitation or volatilization of groundwater which may bias volatile and semi-volatile analyses, and
- 3. may introduce dirt through scraping the sides of the casing wall.
- d. Though bailers are not recommended for purging, they are acceptable if constructed of the appropriate material and if extreme care is used.
- e. All standing water around the top of the well casing (manhole) shall be removed before opening the well.

4.4.2.2 Pumps

- a. Above-ground Pumps
 - Peristaltic Pump Peristaltic pumps may be used to purge low volume, low specific capacity wells in which the static water level in the well is no greater than 20-25 feet BLS (Below Land Surface).
 - a) Decreased pumping velocity will be experienced when water levels are deeper than 18'-20'.
 - b) It also may be used to sample wells for limited parameter groups. These parameter groups will be dependent upon tubing materials and arrangements. It is the preferred method of collecting filtered groundwater samples for metals. See Table A-1 for details on the restrictions for this pump, including choice of tubing (i.e. Teflon, HDPE, Tygon).
 - Centrifugal Pump Centrifugal pumps can be utilized to purge 2 inch and larger internal diameter wells that have moderate specific capacities from 2 10 gpm (gallons per minute) and have a static water level greater than 20 feet BLS.

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- a) The pump may also be attached directly to 3/4" well point casing and used to purge (care must be taken so that purged water does not fall back into the well casing).
- b) Sampling gloves shall be worn and discarded after positioning the pump. Hands should be washed and new gloves shall be put on prior to sampling.
- c) See Table A-1 for compatibility restrictions related to choice of tubing and allowable parameter groups.

b. Submersible Pumps

- 1. Electric Submersible Pumps Submersible pumps (e.g. Grundfos, Goulds, Jacuzzi) can be utilized for purging 4 inch or greater diameter monitor wells. Some submersible pumps can be utilized in 2 inch wells (e.g. Fultz and Grundfos). These pumps can be used in wells that have moderate to high specific capacity and cannot be purged using an above-ground pump because of the lower static water level elevation (>20'-25' BLS).
 - a) The pump must be constructed of stainless steel (and/or Teflon) material and the delivery hose shall be constructed of appropriate material depending upon the analytes of interest.
 - b) It may be fitted with inert stainless steel or Teflon tubing between the pump and "other non-inert tubing" to be able to purge wells that will be sampled for trace organics.
- 2. Bladder Pumps Positive-displacement bladder pumps (no-gas contact) can be utilized for purging wells where the water table is greater than 25 feet and an above-ground pump cannot be used. These pumps are used in wells with low to moderate capacity since pumping rates are not as high as the electric submersibles or the

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gas-contact "purge pump" described below. Maximum pumping rates are approximately 0.5 - 1.5 gallons per minute depending upon the location of the pump (BLS).

- a) The bladder pump system is composed of three major components: the pump, the compressed air and water discharge tubing, and the controller/compressor.
- b) The pump consists of a bladder and an exterior casing or pump body that surrounds the bladder. These two parts can be composed of various materials, usually combinations of PVC, Teflon, and stainless steel.
- c) The construction material of the pump body, pump bladder, and the discharge tubing will define the parameters that can be purged and sampled with this system.
- d) If the pump is not permanently installed in the monitor well and if it is to be used to purge and/or sample for all parameters (including VOCs), the pump, bladder and tubing must be constructed of stainless steel and Teflon.
- e) Permanently installed pumps have a PVC pump body as long as the pump remains in contact with the formation water. If VOCs and/or extractable organics are of interest, the bladder and the delivery tubing shall be constructed of teflon.
- 3. Bladderless Purge Pumps These pumps are identical to the bladder pumps described above except they do not have an internal bladder. The air controller/compressor is used to force water from within the pump body up the discharge tubing. By not having the (Teflon) bladder fill by head pressure, pumping rates are much higher (>4 gpm).

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- a) This pump can only be used for purging.
- b) Additionally, operation of this pump cannot result in purge water escaping back into the well. Proper operation and maintenance of the check valve must be ensured. Release of aerated purge water into the water column is not acceptable.

c. Hand Pumps

- Hand pumps (e.g. Brainard-Kilman 'B-K Pump') are manual pumps
 that should be utilized for purging 2- or 4-inch diameter monitor
 wells in which the static water level is too deep for use of a
 centrifugal or peristaltic pump.
 - a) The B-K hand pump and the associated riser pipes are constructed of PVC and shall be used to purge when only inorganic constituents are of interest unless the restrictions specified in Table A-1 are followed.
 - b) The lower most section of the B-K pump is equipped with a foot valve to prevent back flow of purge water.
 - c) After purging has been completed, the B-K pump should be completely disassembled and decontaminated.
 - d) Please see Table A-1 for details on the use of this pump.

4.4.2.3 Bailers

- a. As stated above, the use of bailers is not recommended for purging.
- Bailers shall be composed of material compatible with the analytes of interest. See Table A-1 for restrictions
 - Bailers constructed of stainless steel and Teflon may be used to sample any and all parameters.
 - 2. Bailers constructed of high density (rigid) polyethylene (HDPE)

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materials may be used to sample monitor wells for inorganics and free-product only.

- 3. When sampling grossly contaminated tanks or other facilities, disposable polyethylene (or other material) bailers should be utilized (it may be difficult to decontaminate such grossly contaminated bailers and as such they may have to be discarded).
- c. The bailer must be handled carefully so as not to contaminate it prior to use.
- d. They shall be scrupulously cleaned, including all check valves.

4.4.2.4 Lanyards

- Lanyard may be disposable (braided or monofilament nylon or reusable (stainless steel or teflon-coated).
- b. A disposable lanyard must be changed for each monitor well, but the same lanyard may be used for purging (if performed) and sampling operations without decontamination between purging and sampling operations.
- c. Reusable lanyards shall be decontaminated between monitor wells but do not require cleaning between purging and sampling operations.

4.4.3 Water Level and Purge Volume Determination

Prior to sampling, an adequate amount of stagnant well water in the well must be removed in order to sample representative formation water. Inspect the exterior protective casing monitor well for damage and document accordingly.

a. Water Level Measurements

1. In order to calculate the purge volume, the water level is determined by using an electronic probe, chalked tape, etc.

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- 2. The depth below land surface shall always be recorded to the nearest 0.1 foot from the same reference or survey mark on the well casing.
- 3. Measurements using an electronic probe shall follow the manufacturer's instructions. Since false reading may be obtained if the sensor contacts the well casing, multiple readings shall be taken to ensure accuracy.
- 4. Decontaminate all measuring devices immediately after use and prior to next measurement.

b. Water Column Determination

- 1. The total water column is obtained by subtracting the depth to the top of the water column from the total depth of the well.
- 2. Total depth of well is dependent upon the well construction. Some wells may be drilled in areas of sinkhole or karst formations. In cases where there may be an open borehole below the cased portion, an attempt should be made to find the total borehole depth.

c. Well Water Volume

The length of the water column is then converted to volume of water that is present in the well:

1. 2 inch casing:

$$V = 0.17 X h$$

Where: V = volume in gallons

h = height of the water column in feet

2. 4 inch casing:

$$V = 0.66 X h$$

Where: V = volume in gallons

h = height of the water column in feet

3. For other casing sizes, calculate using the following:

$$V = (0.041)d X d X h$$

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Where: V = volume in gallons

d = well diameter in inches

h = the height of the water column in feet

or:

V = Pr X r X h(0.001)

Where: V = volume in liters

P = 3.14159 (pi)

r = radius in centimeters

h = height of water column in centimeters

d. Record all measurements in the field records.

4.4.4 Detection And Sampling Of Immiscible Layers

4.4.4.1 Scope/Applications

This procedure covers the methods used to detect and sample immiscible layers. If a facility has a release containing chemical constituents that are insoluble and that have special gravities either greater or less than that of water, then that facility's sampling and analysis plan must address immiscible layers.

4.4.4.2 Summary of Method

The presence of organic vapors should be determined by the use of either a photoionization analyzer or a organic vapor analyzer. The presence of organic vapor may indicate a floating layer on the surface of the groundwater. An interface probe is used to determine the existence of a floating layer. A bailer is then used to sample the floating layer. If a sinker exists a double valve bailer is lowered to the bottom of the well in order to

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sample the layer.

4.4.4.3 Comments

Sampling of the immiscible layer must take place prior to purging. If the floating layer is greater than 2 feet thick then a bottom valve bailer should be used. If the floating layer is less than 2 feet thick but less than 25 feet from the surface then a peristaltic pump should be used. If the floating layer is less than 2 feet thick but more than 25 feet from the surface then a open top closed bottom bailer should be used. A double valve bailer should be used for immiscible layers that are sinkers. Detection and sampling of immiscible layers must be done prior to purging.

4.4.4.4 Procedures

- a. Sample the air in the well head for organic vapors using either a photoionization analyzer or an organic vapor analyzer, and record measurements.
- b. Determine the static liquid level using a water level indicator and record the depth in the log book.
- c. Lower an interface problem into the well to determine the existence of any immiscible layer(s), light and/or dense.
- Remove clean bailer from protective covering, attach cord, type of bailer used will be determined by immiscible layer being sampled. (See comments)
- e. Lower bailer slowly to the interval from which the sample is to be collected. if the sample interval is a floating layer only a few inches thick then the open top bailer should be lowered to the top of the immiscible layer and an additional half thickness of the immiscible

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layer.

- f. Raise bailer to surface, feeding cord into container, reel or onto clean plastic sheeting. Do not allow bailer cord to contact ground.
- g. Remove the cap from the sample bottle, and tilt the bottle slightly.
- h. Pour the sample slowly down the inside of the sample bottle. Avoid splashing of the sample. Assure that any suspended matter in the sample is transferred quantitatively to the sample bottle.
- Leave adequate air space in the bottle to allow for expansion, except for VOA flasks.
- j. Label the bottle carefully and clearly. Enter all information accurately, and check to be sure it is legible.
- k. Samples will be placed in containers defined according to the need, and then, when appropriate, packed with ice or ice packs in coolers as soon as practical.

4.4.5 Well Purging Techniques

To ensure a representative groundwater sample from a monitor well it is essential that the well be purged prior to sampling. Stagnant water in a well casing may undergo a variety chemical changes due to alterations in the redox potential, pH and leaching of organic compounds from the casing.

- a. Equipment selection shall comply with construction and configuration requirements specified in Table A-1.
- b. A clean protective covering may be placed around the wellhead during purging activities. If this protective covering becomes soiled, ripped, etc. it must be replaced prior to sampling.
- c. The total amount of water must be recorded. Therefore, the volume must be measured during the purged operation. The amount may be determined by:

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- 1. Collecting the water in a graduated container (i.e. bucket); or
- 2. Calculating volume based on pumping rate. Note: the pumping rate may not be constant; take this into account.
- d. Record the time that actual purging begins in the field records.
- e. Purging is considered complete if any one of these criteria are satisfied:
 - 1. three well volumes and subsequent stabilization of field parameters
 - a) Stabilization of field parameters is defined as "consecutive readings within 5% taken at least five minutes apart".
 - b) Even if field parameters have not stabilized after 5 well volumes, purging is considered complete and sampling can begin.
 - 2. five well volumes (field parameters not monitored);
 - 3. at least one fully dry purge.
 - a) It has been suggested that one dry purge may not be adequate and a second dry purge may be necessary.
- f. Except for "low recovery" wells, all wells shall be sampled within 6 hours of purging.
 - "Low recovery" wells or wells that have been purged complete dry may be sampled as soon as sufficient sample matrix is available or up to 10 hours after purging.
 - 2. Wells that have not recovered sufficiently within 10 hours of purging should not be sampled.

g. Lanyards

- 1. All lanyards must be securely fastened to downhole equipment (bailers, pumps, etc.).
- 2. Equipment construction and decontamination shall follow guidelines discussed in Purging and Sampling Equipment above.
- 3. Bailer lanyards must be handled such that they do not touch the ground

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surface.

- h. Low Hydraulic Conductivity Monitor Wells (i.e. wells that can be purged dry)
 - The most straightforward method for removing all of the stagnant water in wells screened in low hydraulic conductivity formations is to install the pump in the screen area and pump the well dry.
 - 2. Although this procedure may allow the atmosphere to contact the area of the aquifer immediately surrounding the well screen, it is the best way to ensure that all the stagnant water has been removed.
 - 3. If required, allow the well to recover and purge the well a second time.
- i. High Hydraulic Conductivity Wells (i.e. wells that cannot be purged dry)
 - For those wells with dedicated purging/sampling systems where the pump is set in the screened area of the well, complete evacuation of the stagnant water column may not be possible.
 - 2. The degree to which the stagnant water column can be replaced by fresh aquifer water will be a function of the aquifer transmissivity and the number of well volumes pumped.
- j. In general, when nondedicated pumps that are used for purging, the purging process should be done with the pump as near to the top of the water column as possible to ensure that no stagnant water remains in the well above the screen after purging.
- k. Peristaltic Pump One end of a length of new or pre-cleaned tubing shall be attached to the pumphead flexible hose and the other end immersed no deeper than one foot into the water column.
- 1. Centrifugal Pumps
 - 1. To minimize cross contamination while purging, fuel driven centrifugal pumps must be placed at least 10 feet from the well head and downwind

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of the well.

- 2. Sampling gloves shall be worn and discarded after positioning the pump. Hands should be washed and new gloves shall be put on prior to sampling.
- 3. The length of suction hose should be situated such that the pump will be withdrawing water from the top of the column.
- 4. If the pump rate exceeds the recovery rate of the well then the hose should be lowered into the well as needed to accommodate the drawdown.
- 5. The suction hose must have a footvalve installed to prevent purge water from re-entering the well.

m. Electric Submersible Pumps

- 1. The pump should be set as near the top of the water column as possible to ensure that all stagnant water in the casing is removed and to minimize the contact area of the delivery hose with water column.
- 2. If the pump rate exceeds the specific capacity of the well then the pump must be lowered to accommodate the drawdown.
- 3. If the pump has a controller, the flow rate may be adjusted to be equal (or nearly) to the well's pumping capacity.

n. Bladder Pumps

- This equipment shall be operated strictly according to the owners/operators manual or sample integrity and representativeness may be suspect.
- 2. After determining water level, position the controller/compressor away from the well and downwind (if fuel powered compressor or generator).
- 3. Attach tubing and lower the pump to a depth of 3 5 feet below the surface of the water.

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- 4. If the pump is positioned too deep all of the stagnant water may not be purged. If positioned too shallow purging time will be slower as the bladder fills under standing head pressure.
- 5. Adjust the pump position to follow the water level drawdown, if necessary.
- 6. It may be necessary to adjust the purging rate so that it is equivalent to the drawdown rate.
- 7. Discharge must be directed into graduated bucket or equivalent to determine the number of well volumes.

o. BK Hand Pump

- 1. For the B-K Pump, the intake is lowered to the top of the water column by attaching additional 5-foot sections onto the pump.
- 2. By changing the stroke of the actuating rod the pumping rate can be made compatible with the well-specific yield.

p. Bailers

- 1. The bailer must be handled carefully so as not to contaminate it prior to use.
- 2. The bailer must be lowered through the well and into the formation water slowly. Allowing the bailer to drop into the formation water with a splash is not acceptable.
- 3. The bailer should be used to pull purge water from the top of the water column so that fresh aquifer water can be pulled in through the screen. This technique shall be performed until the requisite number of well volumes have been evacuated.
- q. All purging activities shall be documented in the field notes.

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4.4.6 Groundwater Sampling Techniques

4.4.6.1 Equipment Considerations

- a. Some pumps may be used for sampling groundwater. All notes and restrictions as defined in Table A-1 and discussed in Purging and Sampling Equipment shall be followed when using pumps to collect samples.
- b. Other than the actual sampling device, intermediate vessels should not be used during the sample collection process. This is especially true of any compound where loss of sample is a problem (O&G, TRPH and VOCs). For all trace compounds, the sample should come in contact with as few surfaces or vessels as possible since excessive handling can result in contamination or sample loss.

c. Dedicated Sampling Equipment

- 1. The use of dedicated equipment is recommended since it significantly reduces the chance of cross-contamination.
- 2. Dedicated is defined as equipment that is to be used solely for one location for the life of that equipment (permanently mounted pump or permanently dedicated bailer).
- 3. All material construction and restrictions from Table A-1 also apply to dedicated equipment. Equipment should be purchased with the most sensitive analyte of interest in mind.

4. Cleaning/Decontamination

- a) Dedicated pumps shall be cleaned prior to installation. They need not be cleaned prior to each use but should be cleaned when they are withdrawn for repair or servicing.
- b) Any permanently mounted tubing need not be cleaned.
- c) Any replaceable or temporary tubing shall be cleaned as

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specified in Section 3.6.

- d) Equipment blanks on dedicated pumps shall be required when the tubing is cleaned or replaced and shall be collected through that portion of the tubing that is accessible.
- e) Dedicated bailers, if stored in the well, must be suspended above the water column and completely decontaminated between sampling events.
 - After sampling is complete, they shall be rinsed with tap water and/or analyte-free water, wrapped to prevent contamination, and stored on- or off-site until the next sampling event.
 - 2) The sampling equipment shall be decontaminated prior to on-site arrival UNLESS the equipment is stored on-site. In the latter case, the dedicated bailer shall be fully decontaminated prior to on-site use.
 - 3) A precleaned equipment blank shall be collected prior to reintroducing the cleaned bailer into the water column.

4.4.6.2 Sampling with Bailer

- a. When a bailer is used for sampling, the integrity of the sample collected is highly dependent upon the sampler's skill and familiarity with proper sampling techniques.
- b. It is recommended that for a particular site only two persons perform sampling to minimize personnel handling variation.
- c. Just prior to sampling, several bailer amounts of sample groundwater shall be collected to rinse the bailer.
 - 1. Discard the water appropriately (see Waste Disposal).

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- 2. This should not be done if the analytes of interest include Oil & Grease, TRPH, etc. As stated earlier, intermediate vessels or sampling equipment are never rinsed if these compounds are to be sampled.
- d. All collection activities shall be done carefully so as to not stir up any sediments.
- e. The following procedure describes general bailing techniques:
 - 1. Field personnel should wear protective gloves.
 - 2. Attach a fresh length of monofilament or braided nylon line to the bailer. Alternately, a precleaned permanent lanyard may be used.
 - 3. The bailer or lanyard must not be allowed to touch the ground during purging or sampling.
 - 4. Lower the bailer slowly and gently into contact with the water so that agitation of the water column is minimized.
 - 5. Attempt to sample from the same depth in the well each time, preferably within or just above the screened zone of the well.
 - 6. Do not allow the bailer to touch the bottom of the well so that bottom sediment is incorporated into the sample.
 - 7. Retrieve the bailer smoothly. Collecting the lanyard between the thumbs of each hand seems to be the preferred method.
 - 8. Discard the first few inches of water in the bailer and fill the appropriate sample bottles so that a minimum of turbulence is created to avoid aeration.
 - 9. Discard the last few inches of water in the bailer.
 - 10. Add preservatives (if necessary), check the pH of all pH-adjusted samples (except VOCs).
 - 11. Attach and/or complete the sample container labels, record

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information in field notes, place samples on wet ice (if required) and protect all samples from sun.

f. Disposable Bailers

- 1. Disposable bailers of the appropriate construction material are available. High density polyethylene (HDPE) bailers are acceptable for all inorganic parameters (and free product thickness).
- 2. Teflon bailers are also available as a disposable for use where organics are of concern.
- 3. Precleaned equipment blanks are required for disposable equipment.

4.4.6.3 Sampling with Pumps

As a general rule, pumps shall not be used to collect samples if organics are of interest. There are two exceptions: 1) use of the peristaltic pump with a trap for EXTRACTABLE organics; and 2) use of an all Teflon and stainless steel bladder pump for all organics.

a. Peristaltic Pump

1. Organics

- a) The container shall be a glass or teflon bottle. The sample container is recommended, however, if an intermediate vessel is used, it shall be decontaminated between wells per Section 3.4.1.
- b) All equipment that contacts the groundwater BEFORE the sample container shall be of Teflon, stainless steel or glass construction, including the transport tubing to and from the sample container, the interior liner of the container cap and all fittings.
- c) Connect the outflow tubing from the container to the influent

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side of the peristaltic pump.

- d) Turn the pump on and allow the container to fill approximately 1/4 full.
- e) Turn the pump off, disconnect the container, rinse the bottle and discard the contents.
- f) Repeat the process a second time.
- g) Turn pump on to fill the container.
- h) If an intermediate container is used, distribute the sample into appropriate containers.
- i) If the sample container is used, discard a small portion of the sample, to allow an air space.
- j) Preserve (if required), label and complete field notes.

2. Inorganics

- a) Inorganic samples may be collected from the effluent tubing, and there are few restrictions on tubing type.
- b) If samples are collected from the pump, all tubing (including the tubing in the head) shall be changed between wells.

b. Bladder Pump

- 1. The flow rate shall be reduced after purging to a smooth, even flow.
- 2. When sampling for VOCs, the flow rate must be reduced to 500 ml/minute (approx. 0.1 gallon/min).

c. Other pump types

- 1. Sampling for INORGANICS ONLY may be conducted with most other pump types (see Table A-1).
- 2. The flow rate during sample collection shall be a smooth even flow.
- 3. All tubing and the pump shall be decontaminated between wells.

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4.4.6.4 Sampling Dissolved Metals

- a. In order to collect a "representative" sample for the purpose of monitoring compliance with groundwater standards for metals, it may be necessary to field filter a sample prior to preservation.
- b. In situations where the static level in the well allows use of a peristaltic pump, the groundwater sample shall be pumped directly from the well through an in-line filter.
 - A disposable, high capacity, 1.0 um (metals only) filter is an acceptable filter for most applications. See Table A-1 for allowable equipment setups.
 - 2. In field use, the filter must first be flushed with 30 50 mls of deionized water or an inert gas to remove atmospheric oxygen.
 - The filter must be inserted on the high pressure side (i.e. on the delivery side) of the peristaltic pump. VACUUM FILTRATION IS NOT ACCEPTABLE.
 - 4. The sample delivery tube must be long enough (greater than 2 feet) such that back-diffusion of oxygen to the filter is negligible.
 - 5. New or precleaned silastic tubing shall be installed in the pump at each monitor well.
- c. In situations where the static water level in the well is too deep for a peristaltic pump to be used directly, there are several alternatives:
 - 1. Groundwater may be sampled with an appropriately constructed bailer. The intake tube of the peristaltic pump is inserted into the full bailer and water pumped through a filter as described above.
 - Any submersible pump of appropriate construction for which the flow rate can be adjusted may be used for water levels below 20'-25'.

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- 3. Pressurized HDPE and Teflon bailers may also be used.
- 4. See the specific section concerning field filtration in Table A-1 for all acceptable alternatives.
- d. It is important that this operation is carried out as rapidly as possible and in such a way that sample agitation and exposure to atmospheric oxygen is minimized. It is for this reason that pouring the sample into any intermediate vessel for subsequent filtration IS NOT allowed. This includes barrel or syringe filters. Once the sample is collected into a sample container, preservation and pH checks should be completed.

4.5 Temporary Well Points

Temporary well points include those drilled with augers as well as those pushed with "direct push" or DPT devices. These types of wells are not permanently installed.

4.5.1 Use

- a. Temporary well points may be used for preliminary investigations and as a screening tool.
- b. If these wells are used to provide formal data, these restrictions apply:
 - 1. Use precleaned equipment as described in Table A-1;
 - 2. Well must be purged of 3-5 well volumes (or dry);
 - 3. Sampling with a peristaltic pump
 - a) Extractable organics shall be collected via an all-Teflon and -glass organic trap configuration;
 - b) VOCs shall not be collected through a pump, but the Teflon pump tubing is allowed to fill via ambient pressure, capped with stopper or gloved finger, carefully withdrawn from the well, and drained into appropriate vials.

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- c) Refer to protocols listed in 4.4.5 and 4.4.6 for specific information on sampling and configuration.
- 4. Sampling with bailers
 - a) In some cases, sampling may be accomplished with a 3/4" bailer.
 - b) All equipment construction restrictions shall be followed.
 - c) Refer to bailer sampling protocols in section 4.4.6.

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5.0 SOLID MATRIX SAMPLING PROCEDURES

This section is concerned with grab and (areal or depth) composite samples from solid matrix (soil, sediment and waste piles). Since similar procedures and equipment exist for soils—and sediments, a general description of sample handling will be discussed.

5.1 General Concerns

- a. Sampling equipment shall be selected based on the type of sample to be collected and the parameters of interest. See Table A-1 for specific requirements.
- b. All equipment shall be decontaminated according to specified protocols in Section 3.0.
- c. All general sampling concerns outlined in this document shall be followed.
- d. Sample container and holding time requirements listed in Section 6.2.3 shall be followed. The sample containers shall be cleaned or obtained according to protocols listed in Section 6.1.1.

5.2 Sample Handling Protocols after Sample Acquisition

General sample handling will fall into 3 main categories; surface, shallow subsurface, and deep subsurface. Each of the three categories will be discussed in general. Once the sample is acquired, the handling procedures are very similar and are described below.

- a. Select the appropriate precleaned sampling device and procure the sample from the desired depth. If using liners to transport the sample to the lab.
- b. Select the required sample container for the parameter group.
- c. Split spoons and Shelby Tubes
 - 1. Breakdown the sampler (split spoon, Shelby tube). This should be done with the appropriate tools.
 - 2. At this time, any portion of the sample that has been disturbed shall be identified, removed with a stainless steel spatula and discarded. (Shelby tube only). Cave

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material and soils retained in the auger which enter the split spoon sampler shall likewise be discarded.

- 3. Slice the sample using a clean, decontaminated stainless steel spatula from the center portion of the corer, split spoon or bucket auger head.
- 4. For VOC analyses, EPA Method 5035 shall be followed.
 - a) For low VOC level soil/sediment samples (0.5 to 200 μ g/kg) perform the following:
 - 1) Slice a portion of the cylindrical sample to expose a "fresh" area of sample.
 - 2) Place 5 grams of sample in a pre-weighed vial with a septum-sealed screw cap that already contains a stirring bar and a sodium bisulfate preservative solution. The sample may be transferred to the vial via a cut plastic syringe or by the Purge-and-Trap Soil Sampler™. Transfer the sample with as little disturbance to the sample as possible.

An alternative to collecting the sample in the pre-preserved vial, would be to collect the sample in an EnCore™ Sampler.

- 3) Screw on the top (or seal the EnCore™ Sampler as instructed by the manufacturer).
- 4) Clean the sample container of any loose soil.
- 5) Store the sample on ice at 4°C. Sample collected with the Encore™ Sampler must be analyzed within 48 hours.
- b) For high VOC level soil/sediment samples (>200 μ g/kg) perform the following:
 - 1) Slice a portion of the cylindrical sample to expose a "fresh" area of sample.
 - 2) Place 25 grams of sample in the pre-weighed vial with a septum-sealed screw cap. Transfer of sample to the vial can be made by using a cut

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plastic syringe or by the Purge-and-Trap Soil Sampler™. Transfer the sample with as little disturbance as possible.

As an alternative, an Encore™ Sampler can be used to collect the sample.

- 3) Snap the top off the glass vial containing 25 ml of methanol and empty the contents of the vial over the sample material. Immediately seal the sample container.
- 4) Clean the sample container of any loose soil.
- 5) Store the sample on ice at 4°C. Samples collected with the EnCore™ Sampler must be analyzed within 48 hours.
- c) High Concentration VOC shredder residue samples without field preservation
 - 1) Fill the glass sample vial/container provided by the laboratory to the maximum extent practical, minimizing air/headspace in the sample container.
 - 2) Screw on the septrum seal and clean vial of any debris.
 - 3) Store the sample on ice at 4°C.
- 5. For other analyses, slice sufficient amount of sample from the center portion of the sampling device and transfer it to a tray of appropriate construction (note restrictions on use in Table A-1).
- d. Bucket Auger, Dredge or Corer
 - 1. Remove the sample from the sampler (bucket auger, dredge, corer) with appropriate (stainless steel, teflon, etc.) tools and place in a stainless steel, glass or aluminum foil-lined tray (note restrictions on use in Table A-1.
 - 2. Remove any portion of the sample that has been disturbed with a stainless steel spatula and discarded.
 - 3. If VOCs are required, fill an appropriate container with aliquots that have been taken from selected areas of the entire sample. Proceed as described in 5.2.c.4

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above.

e. Sample Mixing

- 1. VOCs shall be collected as discussed above before the sample is mixed.
- 2. The sample in the tray shall be homogenized thoroughly:
 - a) Appropriate tools shall be used to mix the sample.
 - b) Homogenize by alternately mixing, dividing, and remixing the sample.
- 3. After thorough mixing, transfer the sample to the appropriate sample container(s) leaving minimal headspace.
- f. Clean the outside of the sample container to remove excess soil.
- g. The container rim should also be cleaned of soil and sand particles so that the lid can be sealed. An improperly sealed container may allow cross contamination from ice melt or petroleum fumes.
- h. Affix sample label, seal (if applicable), and complete the chain-of-custody forms.
- i. Place the sample containers in a clean, plastic sample bag and preserve by placing in wet ice.

j. Liners

- 1. If properly used, liners may be inserted into the sampler and used as the actual sample container.
- Be aware that SW-846 has mandated that all solid samples must be transported in containers that have screw tops. This also means that all container and lid requirements are still in effect.
 - a) For inorganic samples, ends of the liner must be covered with polyethylene, Teflon, or aluminum foil sheeting. The sheeting should be secured by placing an end cap over the sheeting.
 - b) For organic samples, the sheeting must be Teflon or aluminum foil.
 - c) With any sample containerized this way, specific instructions must be sent with the sample so that the laboratory will know how to handle the sample.

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All non-volatile samples must be homogenized by the laboratory prior to analyses. Also, any disturbed portions of the sample should be discarded prior to mixing.

5.3 Composite Soil Samples

The following is not a complete discussion regarding development of a sample compositing scheme nor all available sampling protocols. When a large site area is to be investigated for contamination, it is sometimes advantageous to composite soil or sediment samples and thus minimize the number of samples to be analyzed.

- a. Sample aliquots (of identical size) to be composited shall be placed in a tray of suitable materials (see Table A-1) and thoroughly mixed with a cleaned spoon, spoonula or spatula of suitable materials (see Table A-1). The sample shall be thoroughly blended by mixing, and dividing into sections. Each section shall then be mixed separately. Recombine all mixed sections and mix thoroughly. Repeat sectioning and mixing process to ensure proper homogenization.
- b. The origin and size of each (sub)sample or aliquot that is used to make the composite shall be documented in the field notebook along with the other important sampling details.
 - 1. Although the size of these subsamples is important and should be documented, it is critical that these subsamples be of equivalent size, so that the composite sample is not biased by unequal aliquoting.
 - 2. There is no level of accuracy here; it is dependent upon the size of the aliquots.
 - 3. Aliquoting should be done in a systematic manner.
- c. Clean the outside of the sample container to remove excess soil, affix label, seal (if required), and complete the laboratory transmittal forms.

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5.4 Soil Sampling

- 5.4.1 Surface Soil Sampling ground surface to 6 inches below ground surface
 - a. Leaves, grass and surface debris shall be removed from the area to be sampled using a clean stainless steel spoon or shovel.
 - b. Surface soil samples can then be collected using a precleaned stainless steel scoop or spoon.

5.4.2 Shallow Subsurface Soil Sampling

- a. Shallow subsurface samples may be collected by digging a hole or trench to the required depth with a stainless steel shovel.
- b. Some situations may require a trench or pit to be dug with a backhoe. Depending upon the equipment available at the site or the soil type to be penetrated, this option is acceptable.
 - 1. In these situations, the trench is first dug to the appropriate depth and then the sample is exposed by using one precleaned spoon, spatula, or equivalent to clean away the soil that came in contact with the backhoe bucket and a second precleaned spoon to actually collect the sample for all constituents except for VOC's. EnCore™ Samplers, Purge-and-Trap Soil Samplers™ or a cut plastic syringe shall be used to collect the sample for VOC's.
- c. Alternatively, shallow subsurface soil samples may be collected with 2-4 inch stainless steel bucket auger which would minimize the soil to be removed in order to reach the desired depth. Using this method, a sampling depth of up to 15 feet may be obtained.
 - The bucket auger consists of a stainless steel cylinder with flush welded stainless steel cutting edges. The cutting edges are hardened surfaces, heat treated and sharpened.

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- 2. A soil sample is obtained by pushing and rotating the auger into the soil until the bucket is filled.
- 3. The sample can be removed (with some difficulty) from the bucket by pushing or scraping with an appropriate precleaned stainless steel tool.
- 4. This auger method is useful for obtaining large samples of unconsolidated sediment.
- 5. The device is supplied with 3 foot extension rods.
- 6. Addition of a sleeve may allow an undisturbed soil sample to be obtained.
 - a) The device consists of a standard auger head with a removable noncontaminating sleeve which is inserted into the auger barrel.
 - b) Either a clear butyl acrylate (CAB) plastic sleeve (for inorganic samples) or stainless steel (for organic samples) may be utilized.
 - c) The soil sample is obtained in the normal manner by pushing and rotating the auger into the soil. In this case it is the sleeve which fills with soil. After auger retrieval, the sleeve, which is readily removed from the auger, is capped.
- 7. If the auger hole is prone to collapse, due to low cohesion in some soils, a temporary rigid PVC casing should be inserted into the hole. The casing prevents hole collapse and minimizes cross-contamination between soil zones as the auger is advanced.
 - a) Upon sample collection, the temporary casing (if used) must be removed and the hole filled with the excavated soil.

5.4.3 Deeper Subsurface Soil Sampling

a. A drill rig is normally required if soil samples are taken from boreholes greater than 15 feet BLS (below land surface). There are a number of

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sampling devices used in conjunction with the drill rig for retrieving the samples; Shelby tubes, split spoon samplers and standard core barrels.

b. Shelby Tube Sampler

- 1. The Shelby tube sampler is used to sample unconsolidated soils and consists of a stainless steel tube approximately 30 inches long and 2 inches, or larger, in diameter.
- 2. One end of the tube has edges beveled into a cutting edge. The other end can be mounted to an adapter which allows attachment to the drill rig assembly.
- 3. After drilling to the required depth with an auger or rotary drill bit, a soil sample is obtained through the auger or directly in the borehole.
- 4. The Shelby tube is pushed into the soil using the drill rig's hydraulic ram or manually with a sledge hammer.
- 5. When the tube is retrieved, the soil sample taken from the center and away from the sides, can be transferred into the appropriate container (for VOCs) and/or mixed using a stainless steel spoon handle or spatula when other parameters are of interest.

c. Split Spoon Sampler

- 1. A split spoon sampler, useful for sampling unconsolidated soils, consists of two carbon steel half cylinders (spoons) that fit together to form a tube approximately 2 feet in length and 2 inches in diameter.
- 2. The cylindrical arrangement is maintained by a retaining head and bit rings that screw on at either end of the split spoon.
- 3. The bit ring has beveled edges to facilitate sampling as the split spoon is forced into the ground.
- 4. As with the Shelby tube, either the weight of the drill stem and rods or a mechanical hammer is used to advance the sampler.

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- 5. A catcher device is inserted in the head ring to prevent loss of unconsolidated sample during recovery.
- 6. After retrieving the split spoons, the soils can be withdrawn by unscrewing the bit and head rings and splitting the barrel.
- 7. The top 2 to 3 inches of the sample will be normally disturbed and should be discarded.
- 8. EnCore[™] Samplers, Purge-and-Trap Soil Samplers[™] or a cut plastic syringe shall be used to collect the sample for VOC's and/or transfer the contents into an appropriate tray for mixing and containerizing.

d. Standard Core Barrel

- A standard core barrel is utilized when consolidated samples (such as limestone or dolomite) are to be sampled.
- 2. The core barrel is carbon steel cylinder approximately 3 feet long and 2 inches in diameter.
- 3. The barrel has a removable head ring with small embedded diamonds which allow the device to cut through rock or consolidated soils as the drill rods are rotated.
- 4. The sample core can be retrieved by unscrewing the head ring and sliding the sample into the container.

5.5 Sediment Sampling

5.5.1 General Overview

- Sediment samples are taken from material underlying streams/rivers, lagoons, ponds/lakes, and estuaries.
- b. The actual sampling location is dependent upon project scope.
- c. Sediment samples may be taken as an adjunct to surface water samples.
- d. They may be taken as a compositing series to define water or sediment

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quality in a system.

- e. They may be taken above and below an outfall to document degradation.
- f. Similarly, if stressed shore vegetation or visible surface water contamination is evident, sediment samples may be taken.
- g. Decisions for sample location will not be discussed in this document.
- h. All surface water samples shall be taken prior to any sediment samples.

5.5.2 Sample Collection Protocols

- a. Sediment samples are taken via three groups of equipment: scoops; corers and dredges.
- b. Soil sampling equipment is generally not applicable to sediments because of low cohesion of sample.
- c. Sample location (edge or middle of lagoon), depth of water and sediment, sediment grain size (fineness), water velocity, and analytes of interest all must be considered when choosing equipment.
- d. Stainless steel equipment shall be used if trace contaminants are to be sampled.
- e. Dredges must be used for hard or rocky substrates. They are heavy enough to use in high velocity streams.
- f. Coring device may be used for softer substrates. Coring devices must be used in soft substrate if the fine particles are to be included. Coring devices should be used in quiescent waters.

5.5.2.1 Scoops

- a. Scooping is generally most useful around the margin or shore of the water body.
- b. Stainless steel spoons or grain scoops work very well. The scoops can

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be attached to an extendible pole for obtaining samples several feet from shore or boat.

- c. The sampler may also wade into the water body to obtain a scooped sample.
- d. The sampler must stand facing the direction of flow and approach the location from the downstream direction.
- e. Precautions must be taken not to disturb the bottom prior to scooping.
- f. The sample shall be scooped in the upstream direction of flow.

5.5.2.2 Corers

- a. Coring devices can be easily fabricated from many materials. Although stainless steel, glass or teflon must be used for sampling trace organics, other inexpensive material (PVC, carbon steel, etc.) may be used for demands, nutrients, metals as appropriate.
- b. Some corers are simple "push tubes", whereas other more sophisticated models may be finned, gravity driven devices.
- c. Not only are they useful in sampling fine grain sediments, they can also present or preserve the historical layering of sediments.
- d. Upon descent, water displacement is minimal, which also minimizes the shock wave produced by other equipment (dredges).
- e. The corer is the equipment of choice for fine sediments in static waters, especially trace organics and metals.
- f. Corer diameter, grain size, and sample consistency will determine if the sample will remain in the corer upon withdrawal.
- g. Sample washout can be a problem and there are several ways to reduce or prevent it.
 - 1. The leading edge of the corer can be fitted with a nosepiece or core

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catcher which physically keeps the sample from slipping back out the corer.

- a) The core catcher material must also be compatible with analytes of interest.
- 2. A second option is fitting the top or back end with a check valve which first creates negative pressure on the back of the sample as it is being pulled from the substrate and second, prevents surface water from washing out the top portion of the sample.
- h. The corer shall be rotated as it is pushed in.
 - 1. Rotation should be around its axis, not rocked back and forth.
 - 2. Rotation improves penetration and prevents compaction of the sample as it is pushed to the full length of the corer.
- i. Upon withdrawal from the water surface, a cap shall be placed on the bottom to prevent the sample from sliding out.
- j. The core should then be extruded out into a pan or tray and sample processed as described in Section 5.2 above.
- k. Corers can also be fitted with liners. This is advantageous if a complete core is desired that has not been in contact with the atmosphere. It is also advantageous if the coring device is not constructed of the proper material (e.g. PVC) and one of the analytes requires a sampler of inert construction (glass, SS, or Teflon).

5.5.2.3 Dredges

- a. The three main types are the Peterson, Eckman, and Ponar.
- b. The Peterson and Ponar dredges are suitable for hard or rocky substrates.
 - 1. The Peterson and Ponar are virtually the same, except the Ponar has

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been adapted with a top screen and side plates to prevent sample loss upon ascent. For this reason, the Ponar is the dredge of choice for rocky substrates. These dredges are heavy enough to use in streams with fast currents.

- c. The Eckman is designed for softer substrates of sand, silt, or mud.
- 1. The Eckman is too light to use in fast currents.
- d. Follow the manufacturer's suggestions for setting and operating the weighted messenger devices.

5.6 Waste Pile Sampling

The Remedial Investigation Workplan provides the procedures for discrete and composite samples. Due to the large material found in shredder residue, standard EPA Methods for collecting samples for VOC analysis cannot be utilized. Therefore, high concentration VOC sample analysis for shredder residue samples shall be performed without preservation, consistent with Section 6.1.2 of Method 5035. See paragraph 5.2.4(c) of this plan.

6.0 SAMPLE HANDLING

6.1 Sample Containers

6.1.1 Obtaining Clean Containers

Sample containers shall be cleaned or obtained by one of three protocols:

- a. Purchased from commercial vendors as precleaned containers. The cleaning grades must meet EPA analyte specific requirements. All records for these containers (lot numbers, certification statements, date of receipt, etc.) and their intended uses must be documented; or
- b. Obtained from a subcontracted laboratory with an approved sample cleaning and handling protocols in their Comprehensive QA Plan.
- c. Cleaned and maintained by the organization following all analyte specific container cleaning procedures as follows:

6.1.2 Container Cleaning Procedures

The numbered procedures are described after Table 6-1.

TABLE 6-1

Analysis/Parameter	Cleaning Protocols (in order specified)
Extractable Organics (GC, HPLC, GC/MS and Total Phenols)	1, 2, 4, 6, (5 and 7 optional), 12
Purgeable Organics (VOCs) (GC, GC/MS, TOX)	1, 2, 4, (6 optional, methanol only), 7
Metals (Including Cr and Hg)	1, 2, 3, 4, 8, 12

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Inorganics

(Including Cyanide, Alkalinity, Acidity, Residues, BOD, Color, Surfactants, COD, TOC, Chloride, Turbidity, Sulfate, Bromide, Sulfide, Fluoride, Nutrients and Radionuclides)

1, 2, 3*, 4, 8, 9, 12

(* For nutrients, nitric acid should be replaced by hydrochloric acid, or hydrochloric acid may be used after the nitric acid rinse)

Oil & Grease (and TRPH)

1, 2, 3, 4, (5, 6, 7 optional), 12

NOTES:

- a) New container cleaning procedures may skip steps 1 and 2.
- b) This sheet does not represent all possible cleaning procedures, and deviations may be accepted on a case by case basis.

Cleaning Procedures:

- Wash with hot tap water and a brush using a suitable laboratory-grade detergent.
 Organics- Liquinox, Alconox or equivalents
 Inorganic anions- Liquinox or equivalent
 Inorganic cations- Liquinox, Acationox, Micro or equivalents
- 2. Rinse thoroughly with hot tap water.
- 3. Rinse with 1:1 nitric acid solution.
- 4. Rinse thoroughly with deionized water.
- 5. Rinse thoroughly with pesticide-grade methylene chloride.
- 6. Rinse thoroughly with pesticide grade acetone or methanol (acetone only for Bioassays).
- 7. Oven dry at 103 C to 125 C for at least 1 hour.*
 - * VOC vials and containers should remain in the oven in a contaminant-free environment until needed. They should be capped in a contaminant-free environment

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just prior to dispatch to the field or to field sampling consultants.

- 8. Invert and air-dry in contaminant-free environment.
- 9. Container is rinsed with sample unless container already contains preservative.
- 10. Autoclave containers (the tops of which are covered with aluminum foil and autoclave indicator tape is applied over the top of the container).
- 11. Rinse with 10% HCl followed by a sodium bicarbonate solution.
- 12. Cap tightly and store in a contaminant-free environment until use.

6.1.3 Documentation

- a. Records of packing slips and lot numbers (if ordered) and/or records of cleaning protocols for container lots must be maintained.
- b. Cleaning records shall at a minimum record the following:
 - 1. Cleaning procedure;
 - 2. Lot numbers of reagent solvents and acids;
 - 3. Date of cleaning;
 - 4. Initials of person who cleaned containers;
 - 5. Lot number (date of cleaning may be used);
 - 6. If performed, the results of quality control tests that were run on lot numbers; and
 - 7. Any additional cleaning or problems that were encountered with a specific lot.
- c. Records shall be maintained that link lot numbers (either vendor or internal) to projects and/or clients.

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6.2 Sample Preservation and Holding Times

6.2.1 General Considerations

- a. Proper sample preservation is the responsibility of the sampling team, NOT—the lab providing sample containers.
- b. It is the responsibility of the field team to assure that all samples are appropriately preserved.
- c. "IMMEDIATELY" is defined as within 15 minutes. This pertains to preservation as well as filtration immediately followed by preservation (i.e. dissolved metals, orthophosphate, etc.).

6.2.2 Sample Preservation

- a. Sample preservation shall be accomplished by obtaining prepreserved bottles from an acceptable source or actually adding preservative to the sample in the field:
 - Sample containers obtained from the subcontracted laboratory prepreserved. The laboratory shall supply additional same-source preservatives in suitable containers.
 - 2. Sample containers preserved in the field after sample addition. These preservation protocols shall be followed:
 - a. Preservatives shall be reagent grade or of a higher grade. Unless supported by equipment blanks, the acid for metals shall be suitable for trace metals analysis
 - b. Fresh preservatives shall be obtained from parent stocks prior to each sampling event. Any remaining preservatives that are not in sealed ampoules SHALL NOT be returned to stock, but must be appropriately discarded.
 - c. Preservatives shall be transported to the field in plastic or teflon

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containers unless sealed by the manufacturer in glass ampoules.

- d. Preservatives shall be added with disposable pipettes or premeasured ampoules to each sample container
- e. The same amount of preservative shall be added to the associated equipment blanks
- b. The pH shall be checked on all pH preserved samples (except VOC, O&G, and TRPH) using the following protocol:
 - 1. The effectiveness of required pH adjustment must be checked in the field.
 - 2. Narrow range pH paper shall be used to test an ALIQUOT of the preserved sample.
 - a) Pour a small portion of the sample into disposable container
 - b) Place the pH paper into the container and compare the color with the manufacturer's color chart
 - c) Discard the aliquot appropriately. DO NOT POUR BACK INTO THE SAMPLE CONTAINER.
 - 3. If the pH is acceptable (ex. greater than 10, less than 2, etc.), document acceptability in field records and prepare container for shipment to the laboratory.
 - 4. If the pH is unacceptable:
 - Add additional preservative in measured increments, mix and test
 ALIQUOTS of the sample as described above.
 - b) Continue to add measured increments of preservatives until an acceptable pH has been reached.
 - c) Record the TOTAL amount of preservative that was needed.
 - Additional chemical preservatives used in the field shall be from the same source as the chemical used for original preservation. DO

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NOT REUSE OLD SUPPLIES OF PRESERVATIVES.

- Alternatively, an extra "dummy sample" may be used to test pH. Contents of the containers shall be suitably discarded.
- 6. The same amount of additional chemical preservative shall be added to the corresponding equipment blank (or field blank, if used). NOTE: the maximum amount of preservative that was used to preserve any single sample in the sample set SHALL BE ADDED to the equipment blank.
- 7. Sample pH shall be checked at the following minimum frequencies:
 - a) during the first sampling event at a particular site, ALL samples that are pH-adjusted must be checked, and
 - b) during subsequent visits to a particular site, AT LEAST ONE sample per parameter group that must be pH-adjusted shall will be checked.
 - c) If the frequency of sample collection at a specified location is greater than once per month (i.e. weekly or daily), the pH checks shall be made on AT LEAST ONE sample per parameter group according to the following schedule:
 - 1) Weekly sampling 1 pH check per month
 - 2) Daily sampling 1 pH check per week
 - < 1 sampling episodes per day a minimum of 1 pH check,
 and 1 additional check per 10 sampling episodes.
- c. The organization preparing and preserving the containers must keep all documentation for preservation, consisting of:
 - 1. the grades and lot numbers of all preservation reagents
 - 2. the opening date and expiration date.
 - 3. the specific preservation technique that was used with each sample.

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6.2.3 Holding Times, Container Types and Preservation

Holding time, container type and required preservation for samples shall comply with the following tables:

a. Table A-2 (Table II of 40 CFR Part 136)

6.2.4 Special preservation protocols:

- a. All special preservation protocols outlined in Section 4.2 of the surface water section shall be followed for all aqueous samples.
- b. Samples for Chlorophyll shall be treated as follows:
 - 1. samples shall be filtered in the laboratory within 24 hr. of collection,
 - 2. magnesium carbonate shall be added to the filter while the last of the filtrate passes through the filter,
 - the sample will be either analyzed immediately or frozen for later analysis within 21 days.

6.3 Sample Dispatch

6.3.1 Documentation

Field documentation will consist of, at a minimum, field notes, sample labels and Chain of Custody forms (or sample transmittal forms). These items must contain a minimum amount of information that can be traceable back to the original sampling event. A complete discussion of the mandatory information to be completed in the field are in the QAPP.

6.3.2 Sample Packing and Transport

a. Samples shall be packed such that they are segregated by site, sampling location or by sample analysis type. Sample segregation may follow this segregation scheme or any other that is sensible and well thought out. These

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schemes are dependent upon the levels of contamination present, the number of bottles to be transported, the size of the bottles, etc.

- VOC samples from different locations may be placed into the same cooler to reduce the number of required trip blanks provided that the samples are wrapped or containerized (ziplock bag or metal can) separately.
- 2. Samples in breakable containers shall be packed with materials (i.e. bubble wrap, cans with vermiculite) to avoid breakage.
- 3. Shipping transport containers shall be insulated (if cooling is required).
- Shipping containers shall be sealed with strapping tape or locked to avoid tampering. Tamper-proof seals may also be placed over cooler lid.
- All samples that require thermal preservation shall be packed in thermally insulated coolers with wet ice. Only wet ice shall be used in cooling samples to 4°C. BLUE ICE OR CHEMICAL COOLING PACKS ARE NOT ACCEPTABLE.
- b. Packed samples shall be delivered to the analyzing laboratory by the sampling team or via common carrier.
 - 1. If sent by common carrier, all documentation (transmittal form, bill of lading, analyses order, etc.) shall be sealed and placed inside the shipping container prior to sealing it closed.

6.4 Field Reagent Handling

All reagents, cleaning materials and preservatives that are maintained in the field shall be stored, transported and handled in such a way to prevent and/or minimize contamination.

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The following storage and use protocols shall be observed:

- a. All chemicals that are maintained in-house and transported to the field shall be segregated according to reactivity (i.e. acids, bases, etc.).
- b. If possible, acids should be stored in an acid storage cabinet and solvents should be stored in a vented solvent storage cabinet. If specialized storage is unavailable, all chemicals shall be stored in a well-ventilated area.
- All chemicals transported to the field shall be stored in bottles which will be packed to avoid breakage.
- d. If quantities of reagent chemicals are transferred from the original container, the transport container shall be appropriately precleaned and must be of similar construction type as the original container (e.g. acids and bases may be transported in plastic or teflon containers).
- e. Chemicals shall be segregated from sample containers so as to avoid reaction and accidental contamination.
- f. Acids and bases must be segregated to prevent reaction.
- g. Analyte-free water shall be segregated from solvents to prevent contamination.

6.5 Field Waste Disposal

6.5.1 General Considerations

Field-generated wastes may require segregation and containerization for proper disposal by a commercial contractor. This decision is highly dependent upon which type of work is being conducted and the nature of the waste. In general, these wastes can be categorized as: (1) decontamination wastes, especially waste solvents, (2) waste acids and bases, (3) contaminated purge waters, and (4) calibration standards from field meters.

a. All field investigations will generate some amount of waste material,

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especially groundwater investigations. Boring, developing, purging, and sampling monitor wells will generate soils, waters, and spent reagents that must be handled in a way that will not spread or increase contamination at the site. Activities from other sampling matrices will generate similar wastes.

- These wastes are normally categorized into hazardous and non-hazardous wastes.
 - 1. Hazardous wastes must be disposed of according to any and all applicable Federal (RCRA, CERCLA, etc.), State, County, or municipal regulations.
 - 2. Non-hazardous wastes must also be disposed of appropriately.
- c. Proper handling and disposal of all waste materials should be addressed prior to initiating site work. The following are a list of some materials that will require proper treatment, storage, and disposal. Additionally, these must be segregated by their hazardous or non-hazardous nature:
 - 1. personnel equipment: coveralls, gloves, boots, suits, disposable booties,
 - disposable equipment: ground covers, equipment covers (aluminum foil, plastic garbage bags, etc.), disposable bailers or tubing, broken or unused sample containers, shipping containers, etc.,
 - 3. soil cuttings from drilling or hand boring,
 - 4. drilling mud or fluid,
 - 5. development and purge waters,
 - 6. decontamination wastes: spent solvents and acids, and
 - 7. spent calibration standards for field analytical equipment (field GC, conductance, pH, etc.).

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6.5.2 Decontamination Wastes

- Decontamination and calibration wastes must be segregated and disposed of properly.
- b. Soap solutions and waste tap/DI/analyte-free water can be disposed of on site.
- c. Calibration standards (pH and conductance) may be diluted with spent detergent solution and wash waters and disposed of in sanitary sewer.
- d. Weak acid solutions may be neutralized or diluted and disposed of properly.
- e. Waste solvents shall be handled as hazardous waste and must be collected and transported back to the office or lab to be handled by commercial disposal or recycling contractor. DISCHARGE OR EVAPORATION OF WASTE SOLVENTS ON-SITE, IN ANY AMOUNT, IS NOT ACCEPTABLE. See discussion below on hazardous waste handling.
- f. Concentrated, reagent grade preservative acids and bases shall be transported back to the office, laboratory, or disposed of by commercial contractor.
- g. Field GC standards must be handled as a waste solvents.

6.5.3 Disposal of purged water

- a. Contaminated purge waters must be handled prudently.
- b. If wastewater generated from well development or purging of monitor wells is likely to contain contaminants in excess of the MCL (maximum contaminant level), the water shall be contained on-site in temporary storage (e.g., lined pit, drum, tank or tanker truck) until the waters can be characterized by the appropriate approved analytical method(s).
- c. In many cases it may be possible to directly discharge contaminated purged water on-site, but only if the purged water will infiltrate into the SAME aquifer zone from which it was purged from or into a more contaminated

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aquifer zone.

- d. Additionally, exposure of such purged water must not pose a health risk and the purged water shall not be discharged into any surface water body, unless permitted.
- e. Purged water must be adequately treated (contaminants should not exceed established standards) prior to discharge on-site if the above conditions cannot be met.
- f. Alternatively, purged water may be transported to an off-site facility such as sewage treatment plant/sewer system (some wastewater treatment systems are capable of treating water with total VOC concentrations up to 500 ppm). THE WASTEWATER TREATMENT PLANT OPERATOR MUST BE NOTIFIED AND MUST GIVE APPROVAL BEFORE DISCHARGE CAN OCCUR.

6.5.4 Field Generated Hazardous Waste

Handling, storage and disposal of field-related hazardous wastes are subject to the regulations contained in the Resource Conservation and Recovery Act.

All of the procedures listed in this subsection pertain to the company, field consultant, primary contractor, etc. whomever is performing and is responsible for the field sampling event. For brevity, we will refer to this organization as the field consultant.

Responsibilities

- 1. The field consultant is responsible for all wastes generated on-site as a result of the sampling event, excluding those waste materials already present on-site (contaminated drill cuttings, purge water, etc.).
- 2. It is the responsibility of the field consultant to store, package, label,

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ship and dispose of the hazardous wastes which are generated during the sampling event or project in a manner which ensures compliance with all Federal, State and local laws, regulations and ordinances.

- Responsibility may also be assumed by the property owner. These
 requirements will not specify who is ultimately responsible. This
 decision will be made by the property owner and the primary contractor
 with regard to ALL of the RCRA requirements.
- 4. The field consultant is responsible for the waste if it contaminates the environment; therefore, precautions should be taken to secure all reagents (acids, bases, solvents, etc.) that, if spilled, would be characterized as a hazardous waste (listed in 40 CFR Part 261.30-.33 or if a characteristic waste).

b. Definitions

- 1. A hazardous waste can be defined by any one of the following criteria;
 - a) The waste material is listed in 40 CFR Part 261.30-261.33.
 - b) The material exhibits any of the specified characteristics: ignitability; corrosivity; reactivity or TC toxicity.

2. Classification

- a) Field consultants that generate hazardous waste are put into 3 categories based on the amount of hazardous waste generated monthly. These categories are; 1) conditionally exempt small quantity generator, 2) small quantity generator and 3) full generator.
- b) Conditionally Exempt Small Quantity Generator: A generator who generates no more than 100 kilograms of hazardous waste in a calendar month. (40 CFR Part 261.5)
- c) Small Quantity Generator:

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A generator who generates more than 100 kilograms but no more than 1000 kilograms of hazardous waste per calendar month or generates less than 1 kilogram of acute hazardous waste and accumulates no greater than 6000 kilograms of hazardous waste. (40 CFR Part 262.34)

d) Full Generator:

A generator who generates wastes in excess of 1000 kilograms per calendar month or more than 1 kilogram per month of acute hazardous waste. (40 CFR Part 262.34).

- 3. It is the responsibility of the field consultant to know which category their organization falls under. Since most field consultants will fall into the conditionally exempt small quantity generator category, these requirements are listed below.
- c. Hazardous waste Handling Protocols for Conditionally Exempt Small Quantity Generators
 - 1. These generators may either treat or dispose of hazardous waste in an on-site facility or ensure delivery to an off-site treatment, storage or disposal facility, either of which, if located in the U.S., is:
 - a) Permitted under Part 270 of the federal regulations
 - b) In interim status under Parts 270 & 265.
 - c) Authorized to manage hazardous waste by a state with a hazardous waste management program approved under Part 271.
 - d) Permitted, licensed, or registered by a state to manage municipal or industrial solid waste. *(subject to local regulations).
- d. Hazardous Waste Handling Protocols for Facilities falling into the Small Quantity Generator and Full Generator
 - These organizations must adhere to all regulations pertaining to waste disposal in the Resource Conservation and Recovery Act.

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e. General Disposal/Treatment Considerations:

- Hazardous waste solvents, as identified in the 40 CFR Part 261, shall not be evaporated on-site by pouring onto pervious or impervious surfaces.
 - a) These solvents shall also not be evaporated at the office or lab with or without a fume hood.
 - b) Solvents that evaporate during the actual decontamination process are exempt.
- Acidic and Basic wastes may be neutralized and disposed of via the sanitary sewer if they are not hazardous due to the presence of other constituents*. (*subject to local regulations).

f. Transportation

- 1. There are no special handling requirements for transportation of these wastes back to the office or laboratory.
- 2. There are no requirements for manifesting the waste nor placarding the vehicle (if for small quantities).
- 3. A sample collector shipping samples to a laboratory and a laboratory returning samples to a sample collector must comply with U.S. Department of Transportation (DOT), U.S. Postal Service (USPS), or any other applicable shipping requirements.

g. Storage and Accumulation:

- Hazardous waste storage is limited by quantity and/or accumulation time and must comply with RCRA regulations as specified in the 40 CFR.
- 2. These wastes shall be packaged and separated according to compatible groups (e.g. solvents, acids, etc.).

h. Sample Disposal:

1. Samples submitted to a laboratory for analysis are excluded from

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regulation as hazardous waste under 40 CFR Part 261.4(d) provided the samples are being transported to or from the laboratory, or are being analyzed, are being held for analysis, or are being maintained in custody – for legal reasons.

- 2. Once a decision is made to dispose of laboratory samples, the exclusion provisions of 40 CFR Part 261.4(d) no longer apply.
- 3. Samples that have been identified as hazardous may either be: 1) returned safely to the generator; or 2) disposed of according to applicable RCRA regulations summarized in this document.
- 4. Samples which are determined to be non-hazardous may be subject to local environmental regulations. It will be the responsibility of the laboratory to be familiar with any such local regulations.

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7.0 CALIBRATION PROCEDURES AND FREQUENCY

7.1 INTRODUCTION

This SOP stipulates minimum calibration requirements necessary to ensure that the measuring system is capable of producing acceptable data. Acceptable calibration protocol must involve a demonstration that the instrument or measuring system is capable of acceptable performance at the beginning of the analysis sequence and that initial calibration is still valid after continued system operation.

7.2 GENERAL CONSIDERATIONS

- a. Calibrations must be performed according to all analytical method directives OR as indicated in this document if specifics are not addressed in the cited method.
- b. Analytical method calibration acceptance criteria must be followed or if acceptance criteria are not specified in the method, general criteria presented in this SOP shall be used to verify an acceptable calibration.
- c. Calibration of field instruments shall be performed on a regular basis with records kept on a separate calibration log. The records must indicate the method used to calibrate, the time and date, number of standard(s), resulting meter response, actions taken, and the results of the calibration. The meter: name, model number, and identification number (if applicable) shall be entered.
- d. Maintenance and repair notes shall be made in the maintenance logbook for each meter. If rental equipment is used, a log is not required. However, the origin (i.e. rental company), rental date, equipment type, model number and identification number (if applicable) shall be entered into the field notes or a rental equipment notebook.
- e. Prior to mobilization, the manager of the field crew must verify that all equipment is in proper working condition, calibrated, and that batteries are properly charged.

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f. Field calibration of each meter shall occur daily (if a sampling event occurs over several days), at the first sample site and must be verified throughout the day (as noted in 7.5 below). This will ensure field data of a known quality. All field calibrations and checks shall be noted on field sheets.

7.3 STANDARD RECEIPT AND TRACEABILITY

- a. Records to be retained for primary stock standards must include source, type of standard, date of receipt, lot number (if applicable), expiration date and purity statement.
- b. Records to be maintained for preparation of intermediate standards must include identification of primary standards used, preparation date, methods of preparation (including specific dilution information), preparer identification, concentration prepared and expiration date.
- c. Preparation records for working standards must include identification of primary and intermediate standards used in working standard preparation, date of preparation, method of preparation (including dilutions), concentrations prepared and preparer identification.

7.4 FREQUENCY OF STANDARD PREPARATION AND STANDARD STORAGE

7.4.1 Standard Storage

- a. Standards must be stored according to analytical method guidance or supplier recommendations.
- b. If no method or supplier guidance is available standards must be replaced upon decreased instrument response.

7.4.2 Frequency of Standard Preparation

a. If no method or supplier guidance is available standards must be renewed upon

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decreased instrument response.

- b. It is recommended that all primary standards be held for no longer than one year.
- c. Five days prior to a known sampling event where instruments will require calibration, standards shall be checked to verify their expiration date, and replaced if needed.

7.4.3 Documentation on calibration standards (e.g., buffers, KCl, and other reagents)

- a. At a minimum, the date of receipt, expiration dates (noted on the bottle label), and date of first use shall be noted on the standard container.
- b. Expiration dates must be followed.
- c. If reagents or standards are prepared from stock chemicals, they must be analytical reagent grade or better. Potassium chloride standards must be of primary standard grade.

7.5 MINIMUM QUALITY CONTROL REQUIREMENTS

- a. Once the meter has been calibrated, these checks shall take place at intervals of every 2 wells or 4 hours, whichever comes first, and at the end of the sampling day. For instance: the pH meter will be checked against the pH 7 buffer, thermistors will be checked against field-grade thermometers, conductance meters will be checked against one KCl standard, etc.
- b. If a field meter fails a continuing calibration, a complete initial calibration must be performed. In this way, meter response will be addressed without the need for generating historical precision and accuracy statistics.

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7.6 PH METERS

7.6.1 General Concerns

- a. The pH meter is field calibrated on a daily basis (If a sampling event occurs over several days) at the first site. Since field meters do bump around from site to site, calibration is likely to change.
- b. Calibration may be checked on a weekly basis in the laboratory to ensure the % theoretical slope is not less than 90%, indicating a bad electrode. This should be noted in the calibration records. If % slope cannot be determined on the meter, or the manufacturer's optimum specifications are different, manufacturers recommendation for maintaining optimum meter performance shall be followed.
- c. There are several interferences to keep in mind with pH measurement:
 - 1. sodium at pH > or = 10 can be reduced or eliminated by using a low sodium error electrode;
 - coatings of oils, greases, and particulates may impair the electrode's
 response. The electrode bulb should be patted dry with lint-free paper or
 cloth and rinsed with deionized water. If not, acetone may be used to clean
 very hard to remove films, but must be used sparingly so the electrode
 surface is not damaged;
 - temperature effects on the electometric measurement of pH are controlled by using instruments having temperature compensation or by calibrating the meter at the temperature of the samples;
 - poorly buffered solutions with low specific conductance (<200 umhos/cm)
 may cause fluctuations in the pH readings. Equilibrate electrode by
 immersing in several aliquots of sample before taking pH.
- d. Follow the instructions with each type of pH meter. Use secondary standard buffer solutions (pH of 4, 7, 10) purchased from commercial vendors for calibration. Do not reuse buffers.

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- e. Each meter/electrode system must be calibrated at a minimum of two points, at least three pH units apart, bracketing the expected sample pH. Check historical data for expected pH or use pH paper on an aliquot to estimate.
- f. Under normal conditions a pH measurement should be accurate to +/- 0.1 pH unit.

7.6.2 Calibration and Field Use

- a. Check the battery before mobilizing and turn on the meter when you reach the first facility and allow it to equilibrate to ambient temperature.
- b. Calibrate the meters prior to taking samples:
 - 1. Estimate the sample pH range (e.g., history, operator, litmus)
 - 2. Turn function switch to pH position
 - 3. Select the appropriate buffers to bracket the expected sample pH, either pH 4 buffer and pH 7 or pH 7 and pH 10.
 - 4. Remove the protective cap, rinse the electrode with deionized water (DI) and dab dry with lint-free paper or cloth.
 - 5. Place and swirl the electrode in the pH 7 buffer and turn the calibration knob until the reading is 7.0. Repeat step 4 above.
 - 6. Place and swirl the electrode in the second buffer solution (pH 4 or 10). Adjust the temperature knob until the reading is that of the pH standard. Repeat step 4 above.
 - 7. Measure the temperature of the second buffer solution.
 - 8. Turn the slope indicator until the arrow of the temperature compensator points to the temperature of the buffer. The percent to the theoretical slope should be read from the slope scale. A slope of less than 90% (or one not meeting the manufacturer's specifications) indicates a faulty electrode or contaminated buffer and the problem should be corrected before proceeding.

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- c. After calibration follow these procedures to take a pH reading of a freshly collected sample:
 - 1. Pour enough fresh sample into a pH measuring cup to take a reading and measure its temperature. If it differs more than 2 C from the buffer temperature, adjust for the difference by turning the slope indicator until the arrow to the temperature compensator points to the sample's temperature.
 - 2. Place and swirl the pH electrode in the sample (in the cup) and read the pH value. In the case of low specific conductance and meter drift, add 1 ml of 1M KCl (potassium chloride) solution to each 100 ml of sample, swirl and read pH. Note: to make 1M KCl solution, take 74.55 grams of primary standard grade KCl and add it to a 1 liter volumetric flask. Add DI to the 1 liter line on the flask and mix. Solutions of the appropriate strength may be purchased from commercial laboratory suppliers.
 - Turn the meter off after the last reading, discard the sample in the cup, rinse
 the electrode thoroughly with deionized water and replace the electrode's
 rubber cap.
- d. In lieu of performing duplicate measurements (precision) or independent check standards (accuracy), additional calibration checks will be required. Continuing calibration must be done per the following:
 - 1. After the initial calibration, the pH meter shall be checked against the pH 7 buffer at intervals of every 2 wells or 4 hours, whichever comes first.
 - 2. The meter will also be checked against the 7 buffer after sampling has been completed.
 - 3. If the sampling event takes less than 4 hours, then an initial calibration and a post-calibration check will be adequate.
 - 4. If, during the continuing calibration, the response is greater than .2 pH

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units on either side of 7, then a complete initial calibration must be conducted.

5. All initial and continuing calibrations shall be completely documented in bound notebook or field sheets, including: date/time, standard(s) used, resultant meter response, action taken, and technician initials.

7.7 TEMPERATURE

7.7.1 General Concerns

- a. Temperature determinations can be made with any field-grade mercury-filled, alcohol-filled, or dial-type Celsius thermometer as well as an electronic thermistor.
- b. All thermometric devices shall, at a minimum, be checked annually in the laboratory against a National Institute of Standards and Technology (NIST) precision thermometer.
 - 1. The temperature measuring device should be checked at two temperatures against the NIST precision thermometer.
 - 2. Temperatures should agree within +/- 0.1°C. Make note of the calibration in the calibration records. Note the make, model, and serial number of each thermometer.
 - Thermometers that do not meet the acceptance criteria should be disposed of properly.
 - b) If the difference is shown to be constant (i.e. + 0.5 C) over the thermometer range, the thermometer may be used provided that the difference is documented for 10 degree increments, and the correcting factor is used in all measurements.
- c. Use care and proper cleaning procedures to prevent sample cross-contamination.

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7.7.2 Calibration and Field Use

- a. All field-grade thermometers must have completed the annual check against the NIST-grade thermometer. All thermistors must be calibrated in the field with a field-grade (or NIST-grade) thermometer.
- b. Allow the thermometer or thermistor (always use one which has been properly calibrated) to equilibrate to ambient temperature.
- c. Insert thermometer or thermistor in situ when possible or in a portion of the sample. Swirl and take readings when the mercury column, needle, or read-out becomes constant; record the temperature to the nearest 0.5 C. Read to the nearest 0.1° C for a digital gage.
- d. Continuing calibration must also be performed for thermistors. The thermistor should be checked against the field-grade thermometer at 4 hour intervals and at the end of the sampling day.

7.8 SPECIFIC CONDUCTIVITY METER

Specific conductance is a useful method to approximate the total amount of inorganic dissolved solids. Conventional conductivity devices consist of two or more platinum electrodes separated by a test solution. The major disadvantage with this type of system is the possibility of polarization or poisoning (fouling) of the electrodes. Conductivity systems based on the measurement of inductance or capacitance are also available. The electrodes in these systems are insulated by a layer of glass or other insulating material. System response is less rapid, but problems with fouling and polarization are eliminated. Conductivity varies with temperature. For example, the conductivity of salt water increases 3%/degree C at O°C, and only 2 %/degree C increase at 25° C. Therefore, it is necessary to record temperature with conductivity measurements or to adjust the temperature of the samples prior to making conductivity measurements. Most conductivity meters have temperature compensation.

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7.8.1 General Concerns

- a. Follow the manufacturer's instructions.
- b. Samples are preferably analyzed at 25°C. If not, temperature corrections are made and results reported at 25°C.
- c. With good equipment an accuracy of +/- 1% of the reading is achievable.
- d. Typically a conductivity meter is combined with a thermistor to measure water temperature. The temperature measurements are used for both conductivity and DO corrections.

7.8.2 Calibration and Field Use

7.8.2.1 Laboratory Calibration

- a. The meter should be checked in a laboratory in one of three ways:
 - 1. Follow method specifications;
 - Use two standard potassium chloride solutions of 100 and 1,000 umhos/cm or standards that bracket the range of expected sample conductance; or
 - 3. A single check standard in each range of a multi-range instrument.
- b. If the meter does not read within 1% of the standards, determine what the problem is and correct it before proceeding. Most field instruments read conductivity directly. If the meter does not correct all values to 25° C, calculate corrective factors using the procedure in 7.8.3 below. Record all readings and calculations in the calibration records.

7.8.2.2 Field Calibration

The meter must be calibrated in the field with at least one KCl standard prior to analyzing the first sample. The chosen standard must be close to the conductance value of the real samples.

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7.8.2.3 Field Use

- a. Typically, the conductivity probe is immersed at the same time, depth, and location as the DO probe. Measure the water temperature with the conductivity probe.
- b. If the meter is equipped with automatic temperature compensation, adjust the temperature knob on the conductivity meter to the water temperature and read the conductivity. The conductivity meter has a set of positions which multiply the reading by powers of ten in order to measure the full range of potential conductivities. You will need to set this dial to the correct range in order to take a reading. The reading, with the temperature gauge adjusted properly, reports conductivity measured at 25° C.
- c. Switch the dial to take a salinity reading. Use this reading to adjust the DO meter for salinity, if necessary. This should not be used for reporting salinity as a measured parameter, since the calibration is not directly applicable. It may be used as an estimate for salinity for compensation of a DO measurement.
- d. Continuing calibration must be performed on the conductance meter. The meter should be checked against the one KCl calibration standard at 4 hour intervals and at the end of the sampling day.
- e. Rinse off the probe with deionized water and turn off when finished for the day.

7.8.3 Calculations

a. If the meter does not automatically correct for temperature, or if a probe with a cell constant other than 1 is used, the following formula shall be used to correct the data to 25° C:

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$$K = \frac{\text{Km}(C)}{1 + 0.0191(T-25)}$$

Where: $K = \text{conductivity in umhos/cm at } 25^{\circ} \text{ C}$

Km = measured conductivity in umhos/cm at T degrees C

C = cell constant

T = measured temperature of the sample in degrees C

If the cell constant is 1, the formula for determining conductivity becomes:

$$K = (Km)$$
 $1 + 0.0191(T-25)$

b. Refer to SM 2510B, 17th edition, if other calculations (i.e. determining cell constant, etc.) are required.

7.9 TURBIDITY

7.9.1 General Concerns

- a. Sample cells must be extremely clean and free from significant scratches. Minor imperfections in the glass are effectively masked by applying silicone oil.
- b. Touching the sample cells with bare hands should be avoided. The cells should be lifted by the cap when at all possible.
- c. Before mobilizing, the batteries should be checked. A set of batteries should always be transported to the field. Recalibration is required after replacing the batteries.

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7.9.2 Calibration and Field Use:

7.9.2.1 Quarterly laboratory Calibration

- a. The turbidimeter will be calibrated with formazin standards of 20, 100, and 800 NTU every three months. Standards must be made IMMEDIATELY before use.
- b. Formazin standard solutions will be prepared from a 4000 NTU stock solution. The prepared stock solution is stable for up to one year when properly prepared.
- c. Dilution water will be prepared per the manufacturers directions.
- d. The 20, 100 and 800 NTU standards will be prepared per the manufacturers directions.
- e. Follow the manufacturers directions to calibrate the instrument.
- f. Document the formazin standards used, date of calibration, person performing calibration and any problems which might have occurred in the calibration log.

7.9.2.2 Field Calibration

Gelex cells shall be used for field calibration. The standards for these Gelex cells must be adjusted as the instrument is adjusted due to calibration from the formazin standards performed in the laboratory. Three Gelex samples shall be transported to the field and used for field calibration. The range of these three standards are 0-10 NTU, 0-100 NTU, 0-1000 NTU. The reading of the Gelex standards from the turbidimeter after calibration will be written on the cap of the Gelex cell. Readings from the Gelex standards should be obtained prior to taking the first sample, every 2 wells or 4 hours, whichever comes first, and at the end of the sampling day. If the reading is not within 5% of the previously established value, the instrument should be recalibrated with the formazin primary standard.

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7.9.2.3 Field Use

- a. Fill a clean sample cell to the fill line.
- b. After placing the cap on the cell, wipe the cell with a soft, lint-free cloth to remove water spots.
- c. Apply a silicon oil down the length of the cell and wipe with a soft cloth to obtain an even film over the entire surface.
- d. Put the sample cell in the instrument so the diamond or orientation mark aligns with the orientation mark on the instrument.
- e. Close the lid.
- f. Press the button(s) to obtain the reading. Do not hold the instrument while taking a reading.

7.10 ORGANIC VAPOR METERS

Organic vapor meters may be used to perform qualitative or screening procedures in many different situations. These devices are equipped with either a flame ionization (FID) or a photoionization (PID) detector. The FID ionizes organic molecules via a hydrogen flame, whereas the PID uses a lamp. Lamps with different electron voltage (eV) may be used with the PID to ionize specific groups or classes of organic compounds. For specific lamp applications consult the owners manual.

These meters may be used for ambient air screening at sites for health and/or safety reasons. They can be used for headspace analyses of soil samples to determine "gross contamination", for well placement, or for grid sampling. Calibration and use of these types of meters should be performed after consulting the owners manual. There are several procedures that must be accomplished at a minimum:

a. Calibration must be performed on-site, prior to sampling, it is also suggested that additional calibrations against one span gas be performed at 4 hour intervals and/or

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at the end of the sampling day.

- b. The meter must be zeroed with "zero air" or equivalent. If known to be free from interfering components, ambient air may be used.
- At least one span gas must be used for calibration.
- d. Carbon filters must be used to distinguish between methane and other aliphatic halocarbons (FIDs only).
- e. Background corrections must be made if soil borings or split spoon samples are analyzed in ambient air (unnecessary for headspace samples performed in mason jars under foil).

7.11 CALIBRATION DOCUMENTATION

Records must be maintained to document and verify acceptable instrument or measuring system calibration for each analysis.

- a. Records must be maintained for all standard preparations and working standards must be easily traced to intermediate and primary standards used for preparation.
- b. Acceptable calibration verification (% recoveries, correlation coefficients) must be recorded and easily identified with applicable calibrations.
- c. If calibration acceptance criteria are based on manufacturer's instrument specifications or acceptable recoveries specified by QC check sample suppliers, then records of such activities must be maintained. Such records must be easily accessible and must establish verification of acceptance criteria.

7.12 DEFINITIONS

7.12.1 Mid-Range Standard

A standard in the middle of the linear range of the established calibration curve or a standard concentration in the middle of the expected sample concentration range

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depending on the type of determination to be performed.

7.12.2 Intermediate Standard

A standard prepared from the primary stock standard which is diluted to prepare the working calibration standards.

7.12.3 Working Standards

The standards that are actually analyzed to perform the instrument or measuring system calibration.

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8.0 GROUNDWATER WELL INSTALLATION

8.1 Groundwater Well Construction

Following is a description of the specifications and procedures proposed for construction of the groundwater monitoring wells.

8.1.1 Drilling Methods

A variety of well drilling methods are available for the purpose of installing groundwater monitoring wells. The drilling method shall minimize the disturbance of subsurface materials and shall not cause contamination of the groundwater. Regardless of the drilling method selected, drilling equipment shall be steam cleaned before use and between borehole locations to prevent cross contamination of wells. This site will employ hollow-stem continuous auger drilling or air rotary method. Other methods will be used should conditions require it.

8.1.2 Monitoring Well Construction Materials

Well construction materials shall be sufficiently durable to resist chemical and physical degradation and yet not interfere with the quality of groundwater samples. Materials to be used for well casings, well screens, filter packs, and annular seals are covered below.

a. Well Casings and Screens

ASTM, NSR rated, Schedule 40, 2 inch PVC shall be used for the casing pipe and well screens at this site. It should be understood that since PVC pipe is being selected for casing and screening material there may be the possibility that after installation PVC deteriorating compounds could be present in the groundwater. If these compounds are detected, then it must

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assume that the contaminants are contained in the groundwater sample and not from the well casing or screen unless identical compounds are found in the upgradient wells and can not be attributed to wastes on-site.

Plastic pipe sections must be flush threaded or be amenable to connection by another mechanical method such as stainless steel screws. No solvents or glues should be allowed in well construction. These compounds readily leach organic contaminants into the ground water. All well casings and screens should be steam cleaned prior to emplacement to ensure that all oils, greases and waxes have been removed.

b. Filter Pack and Annular Sealant

The materials used to construct the filter pack shall be chemically inert clean quartz sand. Fabric filters shall not be used as filter pack materials. Natural gravel packs are acceptable provided an appropriate well screen slot size is used.

The materials used to seal the annular space must prevent cross contamination between strata. The materials shall be chemically resistant to ensure seal integrity during the life of the monitoring well and chemically inert so they do not affect the quality of the groundwater samples. A minimum of two feet of certified coarse grit sodium bentonite shall immediately overlie the filter pack. A cement and bentonite mixture, bentonite chips/pellets, or anti-shrink cement mixtures shall be used as the annular sealant in the unsaturated zone above the certified coarse grit sodium bentonite seal and below the frost line. Extending from a little below the frost line to the surface, the cap shall be composed of concrete blending into a mounded cement apron (to direct rainwater runoff away from the well) extending outward three feet from the edge of the borehole.

The untreated sodium bentonite seal shall be placed around the casing

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either by dropping it directly down the borehole or, if a hollow-stem auger is used, putting the bentonite between the casing and the inside of the auger stem. Both of these methods present a potential for bridging. In shallow monitoring wells, a tamping device shall be used to reduce this potential. In deeper wells, it may be necessary to pour a small amount of formation water down the casing to wash the bentonite down the hole.

The cement-bentonite mixture shall be prepared using formation water or potable water and placed in the borehole using a tremmie pipe. The tremmie method ensures good sealing of the borehole from the bottom.

The remaining annular space shall be sealed with expanding cement to provide for security and an adequate surface seal. Locating the interface between the cement and bentonite-cement mixture ½ to 1 foot below the frost line, serves to protect the well from damage due to frost heaving. The cement shall be placed in the borehole using the tremmie method.

A one-quarter inch vent hole provides an avenue for the escape of gas. The protective cap guards the casing from damage and the locking cap serves as a security device to prevent well tampering.

As with drilling machinery, the well casing and screen shall be steam cleaned before use. Filter sands, well sealant materials, and anything else that may influence sample quality shall be free of contamination.

8.1.3 Well Intake Design

The design and construction of the intake of the monitoring wells shall:

- 1. allow sufficient groundwater flow to the well for sampling;
- 2. minimize the passage of formation materials (turbidity) into the well; and
- 3. ensure sufficient structural integrity to prevent the collapse of the intake

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structure.

For wells completed in unconsolidated materials, the intake of a monitoring well shall consist of a screen or slotted casing with openings sized to ensure that formation material is prohibited from passing through the well during development. Screen size shall be selected to retain 90% of the filter pack and 40% of the formation material. Extraneous fine-grained material (clays and silts) that have been dislodged during drilling may be left on the screen, in the filter pack, and in the well water. These fines shall be removed from the screen and surrounding area during development. For quality-control purposes, only commercially manufactured screens or slotted casings shall be used. Field slotting of screens is unacceptable.

Screening with 0.010 inch slots shall be used unless geologic conditions discovered at the time of installation dictate a different size. The annular space between the face of the formation and the screen or slotted casing shall be filled to minimize passage of formation materials into the well. A filter pack of clean, well rounded, quartz sand or glass beads in each monitoring well that is constructed on site is recommended. In order to ensure discrete sample horizons, the filter pack shall extend no more than two feet above the well screen. A different filter pack material may have to be considered should geologic conditions at the time of drilling dictate the need for a different size.

8.1.4 Well Development

After the construction of monitoring wells is completed, natural hydraulic conductivity of the formation shall be restored and all foreign sediment removed to ensure turbidity-free groundwater samples. A variety of techniques are available for developing a well. To be effective, they

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require reversals or surges in flow to avoid bridging by particles, which is common when flow is continuous in one direction. These reversals or surges can be created by using surge blocks, bailers, or pumps. Formation water—shall be used for surging the well. Should a well be constructed in low yielding water-bearing formations, an outside source of water may be introduced into the well to facilitate development. In these cases, the water shall be chemically analyzed to ensure that it cannot contaminate the aquifer. If compressed air is used in the development of wells there is the possibility that trace contaminants may be introduced. Therefore, sufficient precaution shall be taken to prevent introduction of contaminants which may be cause for concern. All equipment used to develop a well shall be steam cleaned prior to its introduction into the well.

8.1.5 Documentation of Well Design and Construction

The following information shall be required in the design and construction of wells:

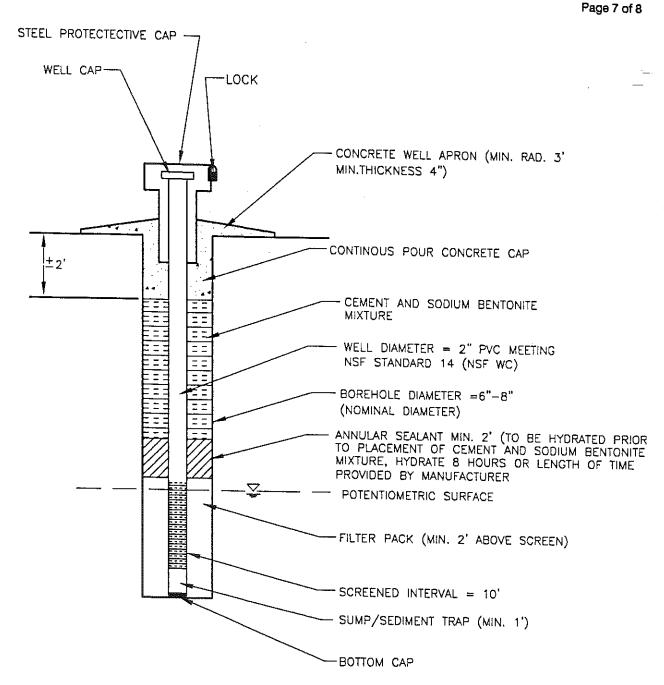
- name of driller, identification of drill rig;
- date of construction;
- drilling method and drilling fluid * (primarily drilling mud) used;
- well location (\pm 0.5 ft.);
- borehole diameter and well casing diameter;
- well depth (\pm 0.1 ft.);
- drilling and lithologic logs;
- casing materials;
- screen materials and design;
- casing and screen joint type;
- screen slot size/length;

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- filter pack material/size;
- filter pack volume;
- filter pack placement method;
- sealant materials;
- sealant volume;
- sealant placement method;
- surface seal design/construction;
- well development procedure;
- type of protective well cap;
- ground surface elevation (± .01 ft.);
- well cap elevation (\pm .01 ft.);
- top of casing elevation (\pm 0.1 ft.); and
- detailed drawing of well (include dimensions).

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FIGURE 8-1



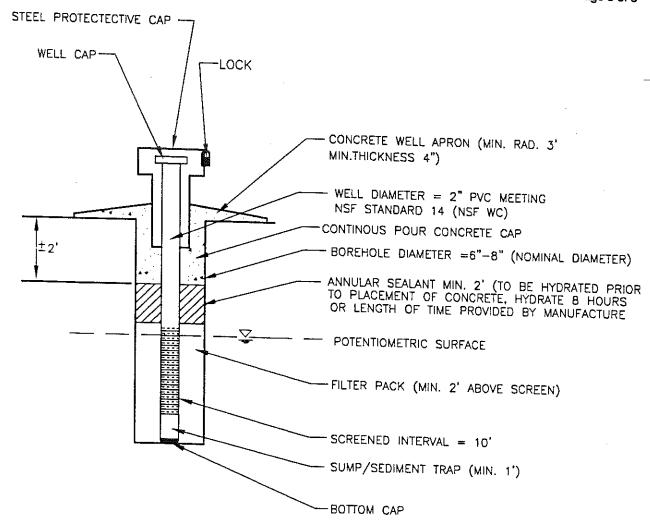
NOTE: SEE FIGURE 8-2 FOR SHALLOW WELL CONSTRUCTION DETAIL (GROUNDWATER LESS THAN 5' FROM GROUND SURFACE)

PROPOSED GROUNDWATER WELL CONSTRUCTION

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FIGURE 8-2

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PROPOSED SHALLOW GROUNDWATER WELL CONSTRUCTION

(GROUNDWATER LESS THAN 5' FROM GROUND SURFACE)
N.T.S.

9.0 GROUNDWATER WELL ABANDONMENT

9.1 Procedure

If it becomes necessary to abandon a monitoring well, the following procedures shall be used. Without proper methods, the abandoned monitoring well may become an avenue of aquifer contamination. Plugging the well can serve to inhibit water loss from artisan aquifers and to eliminate the physical hazard of an open hole. The general procedure for removing and plugging shallow monitoring wells completed in water table aquifers are outlined as follows:

- a. The protective cover and concrete plug shall be removed.
- b. A 3-1/4 inch or larger inside diameter hollow core auger shall be placed over the
 2 inch diameter well casing and advanced to the bottom of the original borehole.
- c. The well casing and screen shall be removed from the auger if possible; if not, the auger shall be removed with the well casing and screen lodged within its core.
- d. If not removed at this point, the auger shall be removed from the borehole.
- e. A tremmie pipe shall be lowered to the bottom of the borehole; if excessive caving has occurred, the tremmie pipe shall be removed, the hollow core auger shall be returned to the borehole, and the tremmie pipe shall be lowered to the bottom of the auger's hollow core.
- f. Cement/bentonite grout shall be pumped through the tremmie pipe to the bottom of the borehole. As the borehole is filled with grout, the tremmie pipe (and auger, if in the borehole) shall be gradually removed. The tremmie pipe shall remain below the grout surface at all times, thus ensuring grout continuity and an effective seal.
- g. The upper 1 foot of the borehole shall be plugged with concrete to form an effective and durable cap.
- h. The materials removed from the borehole, unless otherwise directed, shall be considered to be non-hazardous.

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9.2 Sealant Materials

Well sealants shall be chemically inert and impermeable. Neat portland cement (with or without bentonite clay additives) and bentonite clay are acceptable sealants. General purpose (Type I) neat portland cement is acceptable. The cement slurry is to be mixed with five to six gallons of water for each 94 pound sack of cement. The water of the cement slurry should have a low sulfate content and a total dissolved solids content less than 2,000 parts per million. No aggregate materials are to be included in the slurry.

Bentonite clay additives reduce shrinking (and cracking) of the cement while the slurry is setting. Three to five pounds of additive and 6-1/2 gallons of water shall be mixed with each 94 pound sack of cement (the clay and water are to be mixed together before cement is added to form the slurry). Bentonite clay cannot be used as a sealant where organic contaminants are present in groundwater unless the bentonite is treated and documentation is presented to show that it is capable of containing organic contaminants.

APPENDIX A

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TABLE A-1

RESTRICTIONS AND PRECAUTIONS		a,b; in-line check valve required a,b; in-line check valve required	a,b; in-line check valve required; polishing	none tubling non-metallic if not SS
PERMISSIBLE PARAMETER GROUPS		All parameter groups All parameter groups (excluding VOCs)	All parameter groups	Demands, Nutrients, Metals, Radiochemistry
USE		Purging Sampling	Purging	Sampling
TUBING		SS, Teflon	Non-inert⁴	
CONSTRUCTION		SS, Teflon	SS, Teflon	
EQUIPMENT	WATER SAMPLING Groundwater	Positive Displacement Pumps a. Submersible³ (turbine, helical rotor, gear driven		

HDPE, ETC.) IS NOT RECOMMENDED.

Restrictions/precautions listed on the last page of this chart.

This category refers to tubing and pump housings/internal parts that are in contact with purged or sampled water.

Submersible pumps may be used for purging or sampling only if no other pumping device is available, since lines (power cords, gas pressure tubing) may not be (practically) constructed of inert materials.

[&]quot;Polishing": When purging for organics, the entire length of tubing or portion of which comes in contact with the formation water should be constructed of teflon or stainless steel. If other materials (i.e., PVC< HDPE, or polypropylene) are used, the following protocols must be followed: 1) contact with formation waters is minimized by slowly withdrawing the pump from the water column during the last phase of purging, thus removing from the well any water which may have contacted the exterior of the pump and/or tubing, 2) a single well volume must be removed with the sampling device before sampling begins. Tygon must never be used for purging when organics are of interest. NOTE: THE USE OF NON-INERT (I.E. PVC, "Non-inert" pertains to materials which are reactive (adsorb, absorb, etc.) to the analytes being sampled. Materials include: polyethylene, PVC, and other plastics if organics are of interest and metallic equipment (brass, galvanized, and carbon steel, etc.) if trace metals are of interest.

TABLE A-1 (Continued)

				(22222222)	
EQUIPMENT	CONSTRUCTION HOUSING ²	TUBING	USE	PERMISSIBLE PARAMETER GROUPS	RESTRICTIONS AND PRECAUTIONS
	Non-inert	Non-inert	Purging	All parameter groups	a,b; in-line check valve required; polishing required
			Sampling	Demands, Nutrients, Metals, Radiochemistry	none tubing non-,metallic if not SS
b. Bladder Pump (no gas contact)	SS, Teflon	SS, Teflon	Purging Sampling	All parameter groups All parameter groups	a,b a,b, bladder must be Teflon is sampling for organics
	SS, Teflon	Non-Inert³	Purging	All parameter groups	a,b; polishing required; this configuration <u>not</u> recommended
			Sampling	Demands, Nutrients Metals, Radiochemistry	none tubing non-metallic if not SS
Section 19	Non-inert³	Non-inert³	Purging Sampling	All parameter groups Demands, Nutrients Metals, Radiochemistry	a.b; polishing required None housing & tubing non-metallic if not SS
a. Centrifugal	N/A	SS, Teflon	Purging	All parameter groups	b; foot-valve required
	A/Z	Non-inert ³	Purging	All parameter groups	b; foot-valve required; polishing required

Сį

Restrictions/precautions listed on the last page of this chart.

This category refers to tubing and pump housings/internal parts that are in contact with purged or sampled water.

[&]quot;Non-inert" pertains to materials which are reactive (adsorb, absorb, etc.) to the analytes being sampled. Materials include: polyethylene, PVC, and other plastics if organics are of interest and metallic equipment (brass, galvanized, and carbon steel, etc.) if trace metals are of interest.

TABLE A-1 (Continued)

EQUIPMENT CONSTRUCTION TUBING USE PERMISSIBLE PARAMETER GROUPS RESTRICTIONS AND PRECAUTIONS! WATER SAMPLING Groundwater Groundwater Foot-valve or continuous pumping required Pumps, cont. b. Peristaltic N/A SS/Teflon Purging All parameter groups b; foot-valve or continuous pumping required b. Peristaltic N/A SS/Teflon Purging All parameter groups b; foot-valve or continuous pumping required b. Peristaltic N/A SS/Teflon Purging All parameter groups b; medical grade silicone tubing in pump head
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Restrictions/precautions listed on the last page of this chart.

This category refers to tubing and pump housings/internal parts that are in contact with purged or sampled water.

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TABLE A-1 (Continued)

TUBING USE PERMISSIBLE PARAMETER GROUPS			Specific Depth Grab All parameter groups Sampling	Water Column All parameter groups Composite Sampling	Grab Sampling All parameter groups	Grab Sampling Demands, Nutrients Metals, Radiochemistry	Dissolved Demand, Nutrients constituents Metals in groundwater and static wastewater and No intermediate vessels; positive pressure surface water HDPE & Teflon bailers acceptable
N.	HOUSING ²		ent)SS, Teflon or Teflon-Coated	SS, Teflon or Glass	SS, Teflon	Non-inert³	N/A
EQUIPMENT	WATER SAMPLING	Surface Water	Nansen, Kemmerer, Vandorn (or equivalent)SS, Teflon or Teflon-Coated	2 DO Dunker	3 Bailers		Field Filtration Units

Restrictions/precautions listed on the last page of this chart.

This category refers to tubing and pump housings/internal parts that are in contact with purged or sampled water.

[&]quot;Non-inert" pertains to materials which are reactive (adsorb, absorb, etc.) to the analytes being sampled. Materials include: polyethylene, PVC, and other plastics if organics are of interest and metallic equipment (brass, galvanized, and carbon steel, etc.) if trace metals are of interest.

TABLE A-1 (Continued)

RESTRICTIONS AND PRECAUTIONS	e'p'ɔ	none d		VOC samples may not be taken from composite samples	none must be nonmetallic if not SS	v	W
PERMISSIBLE PARAMETER GROUPS	All parameter groups	Demands, Nutrients Metals, Radiochemistry	All parameter groups	All parameter groups except VOCs	Demands, Nutrients Metals, Radiochemistry	All parameters groups	All parameter groups except VOCs
<u>USE</u>	Sampling	Sampling	Sampling	Compositing	Sampling and Compositing	Sampling	Compositing or homogenizing
TUBING	,						
CONSTRUCTION <u>HOUSING²</u>	SS, Teflon, Glass, Teflon- coated, Aluminum	Non-inert³	SS, Teffon or Teffon-			SS, Teflon, Glass, Teflon Coated or Aluminum	
EQUIPMENT SOIL SAMPLING Sediments/Soils	1 Core Barrel (or liner)		2 Trowel, Scoop, Spoon or Spatula			3 Mixing Tray (pan)	

Restrictions/precautions listed on the last page of this chart.

This category refers to tubing and pump housings/internal parts that are in contact with purged or sampled water.

TABLE A-1 (Continued)

RESTRICTIONS AND PRECAUTIONS	s none e; must be nonmetallic if not SS	попе	none	o,d	O	c,d; samples for VOC and Metals must be taken from the interior of the core sample
PERMISSIBLE PARAMETER GROUPS	Compositing or homogenizing Demands, Nutrients none Metals, Radiochemistry	All parameter groups	Demands, Nutrients	All parameter groups	All parameter groups	All parameter groups
USE		Sampling	Sampling	Sampling	Sampling	Sampling
TUBING						
CONSTRUCTION HOUSING	Non-inert	SS	Non-SS	SS or carbon steel w/ teflon insert	SS	Carbon steel
EQUIPMENT SOIL SAMPLING Mixing Tray, Cont.		4 Shove, Hand auger, Bucket Auger		5 Split spoon	6 Shelby tube	

Key to Restrictions/Precautions

- If used as a non-dedicated system, pump must be completely disassembled, if practical, and cleaned between wells. Delivery tubing must be precleaned and precut at the base of operations or laboratory. If the same tubing is used during the sampling event, it must be cleaned and decontaminated between
- If samples are sealed in the liner for transport to the laboratory, the sample for VOC analysis must be taken from the interior part of the core.

 Liners must be constructed of stainless steel or a suitable non-metallic material. If a metallic (carbon steel, aluminum) liner is used with the core barrel, the samples for metals shall be taken from the interior part of the core sample.

 Aluminum foil, trays or liners may be used only if aluminum is not an analyte of interest. o o

"Non-inert" pertains to materials which are reactive (adsorb, absorb, etc.) to the analytes being sampled. Materials include: polyethylene, PVC, and other plastics if organics are of interest and metallic equipment (brass, galvanized, and carbon steel, etc.) if trace metals are of interest and metallic equipment (brass, galvanized, and carbon steel, etc.) if trace metals are of interest.

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Acronyms:

not applicable stainless steel high density polyethylene polyvinyl chloride volatile organic compound N/A SS HDPE PVC VOC

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TABLE A-2

40 CFR Part 136 TABLE II: REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES (WATER/WASTEWATER SAMPLES)

PARAMETER #	PARAMETER NAME	CONTAINER1	PRESERVATION ² , ³	MAX HOLD TIME ⁴
Table 1A-Bacter	rial Tests:			
1-4.	Coliform, fecal and total	P, G	Cool 4C, 0.008% Na ₂ S ₂ O ₃ ⁵	6 hours
5.	Fecal streptococci	P, G	Cool 4C, 0.008% Na ² S ₂ O ₃ ⁵	6 hours
Table 1B-Inorgar	nic Tests:			
1.	Acidity	P, G	Cool 4C	14 days
2.	Alkalinity	P, G	Cool 4C	14 days
4.	Ammonia	P, G	Cool 4C, H ₂ SO ₄ to pH<2	28 days
9.	Biochemical oxygen demand	P, G	Cool 4C	48 hours
11.	Bromide	P, G	None required	28 days
14.	Biochemical oxygen demand carbonaceous	P, G	Cool 4C	48 hours
15.	Chemical oxygen demand	P, G	Cool 4C, H ₂ SO ₄ to pH<2	28 days
16.	Chloride	P, G	None required	28 days
17.	Chlorine, total residual	P, G	None required	Analyze immediately
21.	Color	P, G	Cool 4C	48 hours
23-24.	Cyanide, total and amenable to chlorination	P, G	Cool 4C, NaOH to pH>12, 0.6g ascorbic acid ⁵	14 days ⁶
25.	Fluoride	Р	None required	28 days
27.	Hardness	P, G	HNO₃ to pH<2, H₂SO₄ to pH<2	6 months
28.	Hydrogen ion (pH)	P, G	None required	Analyze immediately
31, 43.	Kjeldahl and organic nitrogen	P, G	Cool 4C, H₂SO₄,to pH<2	28 days

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TABLE A-2 (Continued)

PARAMETER #	PARAMETER NAME	CONTAINER ¹	PRESERVATION ^{2,3}	MAX HOLD TIME ⁴
Metals ⁷				
18.	Chromium VI	P, G	Cool 4C	24 hours
35.	Mercury	P, G	HNO ₃ to pH<2	28 days
3. 5-8, 10, 12, 13, 19, 20, 22, 26, 29, 30, 32- 34, 36, 37, 45, 47, 51, 52 58- 60, 62, 63, 70- 72, 74, 75	Metals, except chromium VI and mercury	P, G	HNO ₃ to pH<2	6 months
38.	Nitrate	P, G	Cool 4C	48 hours
39.	Nitrate-nitrite	P, G	Cool 4C, H ₂ SO ₄ to Ph<2	28 days
40.	Nitrite	P, G	Cool 4C	48 hours
41.	Oil and grease	G	Cool 4C, H ₂ SO ₄ to pH<2	28 days
42.	Organic carbon	P, G	Cool 4C, HCl or H ₂ SO ₄ to pH<2	28 days
44.	Orthophosphate	P, G	Filter immediately, Cool 4C	48 hours
Table 1C-Organ	ic Tests:8			
13, 18-20, 22, 24-28, 34-37, 39-43, 45-47, 56, 66, 88, 89, 92-95, 97.	Purgeable Halocarbons	G, Teflon- lined septum	Cool 4C, 0.008% Na ₂ S ₂ O ₃ ⁵	14 days
6, 57, 90	Purgeable aromatic hydrocarbons	н	Cool 4C, 0.008% Na ₂ S ₂ O ₃ ⁵ HCl to pH2 ⁹	14 days
3, 4,	Acrolein and acrylonitrile	и	Cool 4C, 0.008% Na ₂ S ₂ O ₃ ⁵ Adjust pH to 4-5 ¹⁰	14 days
23, 30, 44, 49, 53, 67, 70, 71, 83, 85, 96.	Phenois ¹¹	G, Teflon lined cap	Cool 4C, 0.008% Na ₂ S ₂ O ₃ ⁵	7 days until extraction, 40 days after extraction
7, 38.	Benzidines ^{11,12}	н	Cool 4C, 0.008% Na ₂ S ₂ O ₃ ⁵	7 days until extraction, 40 days after extraction ¹³
14, 17, 48, 50- 52.	Phthalate esters ¹¹	и	Cool 4C	7 days until extraction, 40 days after extraction
72-74.	Nitrosamines 11,14	II	Cool 4C, store in dark, 0.008% Na ₂ S ₂ O ₃ ⁵	U
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TABLE A-2 (Continued)

PARAMETER #	PARAMETER NAME	CONTAINER1	PRESERVATION ^{2,3}	MAX HOLD TIME⁴	
76-82.	PCBs 11 acrylonitrile	71	Cool 4C	Į į	
54, 55, 65, 69.	Nitroaromatics and isophorone 11	н	Cool 4C, 0.008% Na ₂ S ₂ O ₃ ⁵ store in dark	IF	
1, 2, 5, 8-12, 32, 33, 58, 59, 64, 68, 84, 86.	Polynuclear aromatic hydrocarbons ¹¹	11	n	U	
Table 1D-Pesticides Tests:					
1-70.	Pesticides ¹¹	*1	Cool 4C, pH 5-9 ¹⁵	u .	
Table 1E-Radio	logical Tests:				
1- 5.	Alpha, beta and radium	P, G	HNO3 TO pH<2	6 months	

Reference: This table is reprinted from 40 CFR Chapter I, Revised as of July 1, 1988. According to Federal Register of Thursday, September 3, 1987, preservation for Oil and Grease may also be performed with HCI

^{1.} Polyethylene (P) or Glass (G).

^{2.} Sample preservation should be performed immediately upon sample collection. For composite chemical samples each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4° C until compositing and sample splitting is completed.

^{3.} When any sample is to be shipped by common carrier or sent through the United States Mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirements of Table II, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid (HCI) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO3) in water solutions at concentrations 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H2SO4) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); and Sodium hydroxide (NaOH) in solutions at concentrations of 0.080% by weight or less (pH about 1.2.30 or less).

⁴ Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. Samples may be held for longer periods only if the permittee, or monitoring laboratory, has data on file to show that the specific types of samples under study are stable for the longer time, and has received a variance from the Regional Administrator under Part 136.3(e). Some samples may not be stable for the maximum time period given in the table. A permittee, or monitoring laboratory, is obligated to hold the sample for shorter time if knowledge exists to show that this is necessary to maintain sample stability. See Part 136.3(e) for details.

^{5.} Should only be used in the presence of residual chlorine.

^{6.} Maximum time is 24 hours when sulfide is present. Optionally all samples may be tested with lead acetate paper before pH adjustments in order to determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to pH 12.

^{7.} Samples should be filtered immediately on-site before adding preservative for dissolved metals.

^{8.} Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.

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- 9. Sample receiving no pH adjustment must be analyzed within seven days of sampling.
- ^{10.} The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within 3 days of sampling.
- When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity. When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to 4 C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6-9; samples preserved in this manner may be held for seven days before extraction and for forty days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (re the requirement for thiosulfate reduction of residual chlorine), and footnotes 12, 13 (re the analysis of benzidine).
- ^{12.} If 1,2-dephenyl hydrazine is likely to be present, adjust the pH of the sample to 4.0 "0.2 to prevent rearrangement to benzidine.
- 13. Extracts may be stored up to 7 days before analysis if storage is conducted under an inert (oxidant-free) atmosphere.
- ^{14.} For the analysis of diphenylnitrosamine, add 0.008% Na2S2O3 and adjust pH to 7-10 with NaOH within 24 hours of sampling.
- 15. The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008% Na2S2O3.

TABLE A-3

RECOMMENDED SAMPLE CONTAINERS, SAMPLE VOLUMES, PRESERVATION TECHNIQUES AND HOLDING TIMES FOR RESIDUALS, SOIL AND SEDIMENT SAMPLES 1

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PARAMETER GROUP	METHODS	REFERENCES	CONTAINER	PRESERVATION	MAX HOLDING
Volatile Organics	Purge-and-Trap GC and GC-MS	8010, 8015, 8020, 8021, 8230, 8240, 8260	Glass, 40 ml vial or 4 oz. wide- mouth with Teflon/silicone septum ²	3	TIMES 14 days
Semivolatile Organics	GC, HPLC, and GC-MS	8040, 8060, 8080, 8090, 8100, 8120, 8250, 8270, 8310	Glass, 8 oz. widemouth with Teflon lined cap(50 grams sample)	3	14 days until extraction, 40 days after extraction.
Total Metals-except mercury and chromium VI	Flame AA, Furnace AA, Hydride and ICP	All 7000-series methods(except 7195, 7196, 7197, 7198, 7470, and 7471) and 6010 (ICP)	Glass or plastic, 8 oz. widemouth (200 grams sample)	. 3	6 months
Chromium VI	Colorimetric, Chelation with Flame AA	7196 and 7197	Glass or plastic, 8 oz. widemouth (200 grams sample)	3	24 hours
Mercury	Manual Cold Vapor AA	7471	Glass or plastic, 8 oz. widemouth (200 grams sample)	3	28 Days

^{1.} Adapted from tables 3-1 and 4-1 in Test Methods for Evaluating Solid Waste, SW-846, EPA, Third Edition, 1986, and First Update in 1987. The term residuals includes: (i) concentrated waste samples and (ii) sludges of domestic or industrial origin.

² Sample shall not be homogenized (mixed) prior to filling container. Container must be filled by packing as much sample into it leaving minimal headspace. Field samples can not be composited for analysis.

³ Soils, sediments and sludges shall be kept cool at 4° C from collection time until analysis. No preservation is required for concentrated waste samples.